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PROCEEDINGS

of the

FLAME RETARDANT COTTON BATTING WORKSHOP

held at

New Orleans, Louisiana
November 15, 1968

Agricultural Research Service

UNITED STATES DEPARTMENT OF AGRICULTURE

PREFACE

The Flame-Retardant Cotton Batting Workshop was sponsored jointly by the Foundation for Cotton Research and Education of the National Cotton Council of America, the National Association of Bedding Manufacturers, the National Cotton Batting Institute, the National Cottonseed Products Association, Textile Fibers and By-Products Association, and USDA's Southern Utilization Research and Development Division (SURDD).

The principal purpose of the meeting was to acquaint the cotton batting industry with current developments in research that may lead to acceptable flame retardant bedding and upholstery products for furniture and automobiles. Additionally, general information was presented that should be of interest to manufacturers of especially flame-retardant cellulosic textile products.

This Workshop was held November 15, 1968, at New Orleans, La. The General Chairman was Lippman Lumberg of Allen Industries, Inc. The program was developed by B. H. Wojcik, Assistant Director, under the direction of C. H. Fisher, Director, in cooperation with staff members.

These proceedings report in full the statements presented by the various speakers during the conference and give an account of the discussions following.

The opinions expressed by the participants appearing at this workshop are their own and do not necessarily represent the view of the U. S. Department of Agriculture.

Underscored numbers in parentheses refer to references at the end of each paper. The data presented in the references, figures, and tables are reproduced essentially as they were supplied by the author of each paper.

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CONTENTS

Page

Opening Remarks
L. Lumberg 1

Welcome
C. H. Fisher 2

Response
A. Grehan, Jr. 4

Utilization and Future Needs for Fire Retardant Materials
A. J. McQuade 5

Potential for Cotton Batting Products
G. S. Buck, Jr. 7

FIRST SESSION
J. M. Bing, Chairman

The Flammable Fabrics Art
S. L. Sutcliffe 9

Implementation of The Flammable Fabrics Act
J. V. Ryan 12

Flame Retardant Cotton-state of the Art
L. L. Heffner 16

Theories of Flame Retardancy and Flame Retardant Upholstery Fabrics and Mattress Ticking
G. L. Drake, Jr. 25

Flame Retardant Cotton Flote
P. A. Koenig 29

SECOND SESSION
S. J. Henry, Jr., Chairman

Latest Research at SURDD to Develop Flame Retardant Conventional Cotton Batting
N. B. Knoepfler 40

PANEL DISCUSSION:
AVAILABLE FLAME RETARDANTS

Pyroset Flame Retardant G-2 50

Available Flame Retardants
J. F. Sturgeon 52

A Fire Retardant for Cotton Batting
A. H. Gaede 54

Test Method, Material Development and Testing Laboratory 57

Reactive Fire Retardant Textile Finish
G. Chynoweth 58

Borates for use in Fire Retardancy
J. G. Bower 60

PANEL DISCUSSION:
APPLICATION OF CHEMICALS TO COTTON FIBERS FOR THE PRODUCTION OF FLAME RETARDANT BATTING

Application of Chemicals to Cotton Fibers for Fire Resistant Batting
W. L. Kuechler 62

Various Methods for Fire Retarding Cotton Batting
W. W. Morgenthaler 64

Chlorinated Solvent Systems for Textile Applications
R. H. Symm 70

Attendance List 72

PROCEEDINGS OF THE FLAME RETARDANT COTTON BATTING WORKSHOP HELD AT NEW ORLEANS, LOUISIANA, NOVEMBER 15, 1968

OPENING REMARKS

by

L. Lumberg
Allen Industries, Inc.
Detroit, Mich.

Welcome all of you to this workshop on flame retardant cotton batting. We are delighted that our program has attracted such an excellent attendance.

Members of the United States Department of Agriculture staff here in New Orleans, the Cotton Batting Institute staff, and our program participants have devoted much time and effort to bring to you the latest and best information available about this very important question of flame retardancy.

Briefly, and by way of background, I am sure that interest in flame retardancy was heightened last year with amendment of the Flammable Fabrics Act. This amendment broadened the scope of the existing law to include interior furnishings, about which we will hear more later directly from the Department of Commerce officials who are involved in the administration of this law. Also of interest to many of us in the cotton batting industry is the new attention that Detroit is directing toward flame retardant treatments for automotive interiors in its Docket 3-3 Motor Vehicle Safety Standard. Whatever your own interest in fire retardancy and cotton batting is, I think we can safely say that your questions will be answered here today.

Therefore, on behalf of the National Cotton Batting Institute (NCBI), I would like to express our appreciation to the other organizations that are supporting this workshop and are joint sponsors with NCBI in our research program at the U. S. Department of Agriculture laboratory. They include the Foundation for Cotton Research and Education, The National Association of Bedding Manufacturers, the National Cottonseed Products Association, and the Textile Fibers and By-Products Association.

WELCOME

by

C. H. Fisher, Director
Southern Utilization Research and Development Division
New Orleans, La.

We are sorry that Dr. Fred R. Senti, Deputy Administrator of Agricultural Research Service, can't be with us. He sends his greetings and best wishes.

Research on flame resistance and cotton batting represents an excellent example of how various groups—including industry and government—can work together harmoniously to maximize efficiency and to achieve major successes beneficial to all segments of our society.

Because so many have cooperated in this endeavor, it is appropriate that this Conference is sponsored by several organizations.

I wish to review briefly research in our Southern Utilization Research and Development Division (SURDD) on flame resistance and cotton batting. The SURDD has long had an interest in flame resistance research. The earliest research on flame resistance in the SURDD was initiated in the early 1940's. Although the effort was small, three papers describing the results were published. The first paper describing SURDD research appeared in 1946 under the title "Durable Flameproofing Suitable for Cotton Outer Garments" by K. S. Campbell and J. E. Sands. In 1949 the SURDD published the following two papers: "Composition of Two Types of Cellulose Phosphates" by J. D. Reid, L. W. Mazzeno, Jr., and E. M. Buras, Jr., and "Preparation and Properties of Cellulose Phosphates" by J. D. Reid and L. W. Mazzeno, Jr.

Thus, in the 1940's the SURDD published three papers related to flame resistance, one in 1946 and two in 1949. This means, of course, that the effort was limited.

In the early 1950's the SURDD renewed its flame resistance research. This time the program grew into one of considerable magnitude. This program in one form or another, has remained active and it is still continuing. During the 13-year period 1954-1967, we published 51 papers on flame resistance for an average of four per year.

How did we happen to get back into flame resistance research in the early 1950's? George Buck and some of his colleagues in the National Cotton Council played a major role. George Buck not only argued that flame resistance was important, but also said that he would attempt to get financial support for the work.

As a member of a committee advising the Quartermaster on flame resistance, George Buck recommended that funds be made available to the SURDD for flame resistance research. Indeed, the funds were made available to us by the Quartermaster and the work was initiated. The Cotton Council also has provided some financial support for flame resistance research.

We received more than funds. We received excellent advice and suggestions from many persons, including: George Buck, Leonard Smith, Bill Segall, and others from the National Cotton Council; Stephen Kennedy, Allan McQuade, and others from the laboratories at Natick; Ed Kropa (decreased) of Battelle Memorial Institute; J. C. King of Reeves Bros.; and Ramon Esteve, then at the University of Delaware. Both the funds and advice were exceedingly helpful, and we are grateful for both.

We are grateful also for experimental and testing assistance provided by our friends at Natick. Through laboratory research and product evaluation, the Natick laboratories made additional valuable contributions to the success of the overall program.

I wish also to thank the group in the SURDD working on flame resistance for their invaluable contributions to the success of the project. For many, it was necessary to terminate work on other attractive projects to begin the new work. But this was done, and great dedication, competence, and hard work were devoted to the new program.

Substantial contributions have also been made by several commercial firms, which developed useful manufacturing methods and formulations. Among these are the Hooker Chemical Co. and Lynn-Russ Finishing Co.

This major cooperative effort, propelled by funds, expertise, and creative thinking from various organizations and many individuals, was highly successful.

As you are aware, several attractive methods were discovered for conferring flame resistance on cotton. New chemicals became commercially available. Expressions like "THPC" and "APO" became commonplace in some circles. The new treatments—after some modifications—were and are applied commercially. John Guthrie and Wilson Reeves received the prestigious John Scott Award for their important contributions and leadership in flame resistance research. And last, but not least, the new knowledge from the research has proved useful in flame resistance research in two other areas, namely, cotton batting and protective coatings.

Repeating, our major program on flame resistance was urged upon us, and funded in part, by our friends in technical and trade associations, industry, and the Department of Defense.

The same is true for our research on cotton batting. Several individuals recommended strongly that we initiate research to improve the usefulness and value of cotton batting. These individuals and the organizations provided funds, valuable advice, and assistance in various ways. Again we had—and have—a cooperative program, a program that belongs to all participants.

Early in the program, great progress was made in achieving improvements in resilience and certain related properties. More recently, improvements were made in flame resistance. The result of all this work has been success—success technically, commercially, and economically.

I wish to thank everyone engaged in and facilitating progress in the cotton batting research. These include many persons, among whom are:

Nestor Knoepfler, Henry Vix, and many others in the SURDD;

W. G. Van Saun, Janesville Cotton Mills;

W. J. McEwen, Burkart Manufacturing Co.;

Milton Warren, Standard Cotton Products Co.;

J. C. Barnhart, Barnhart Manufacturing Co.;

Don A. Bryant, Bryant Bros. Textile, Inc.;

Carl McCabe, Milwaukee Bedding Co.;

W. P. Rocker, Southern Cross Industries, Inc.;

I wish to thank the many individuals who have worked for the benefit of this Conference. These include: our speakers and chairmen, and especially Messrs. Sutcliffe and Ryan of the U. S. Department of Commerce, for their participation; the leaders of all organizations supporting and aiding in the development of the program for this meeting; and

Lippman Lumberg, President, National Cotton Batting Institute;

Stanley Henry, Chairman Research, National Cotton Batting Institute;

Julian Bing, Chairman Research, Textile Fibers and By-Products Association;

Arthur Grehan, Executive Secretary, National Cotton Batting Institute;

Albert S. Roistacher, Executive Vice-President, National Association of Bedding Manufacturers;

Garlon Harper, National Cottonseed Products Association;

George Buck, Foundation for Cotton Research and Education of the National Cotton Council of America;

Burruss B. McGuire, Jr., Department Health, Education and Welfare; and

Scientists, engineers, and others in our SURDD.

RESPONSE

by

A. Grehan, Jr.
National Cotton Batting Institute
Memphis, Tenn.

On behalf of the National Cotton Batting Institute and the other industry sponsors, the Foundation for Cotton Research and Education of the National Cotton Council of America, the National Association of Bedding Manufacturers, the National Cottonseed Products Association, the Textile Fibers and By-Products Association, I would like to express appreciation to Dr. Fisher and to the other personnel for the part they have played in making this program available to us.

I will try not to dwell on the direct question of flame retardancy as it relates to cotton batting, or the end products in which it will be used, as there are many others who can do that. I would like to stress that there is more than just luck or good fortune involved in the fact that there is a fire retardant research project to give this workshop a reason for being. We have this current research program under way, thanks to the foresight and alertness of people in the batting industry and their suppliers.

Each of you present has an important stake in what is going to be discussed today. The purpose of this workshop is twofold—first, to have representatives of the Department of Commerce give you an authoritative interpretation of how the Flammable Textiles Act will affect your products, and second, to inform you of the present state of the art of making cotton batting flame retardant. We owe the United States Department of Agriculture our grateful thanks for making this program possible.

UTILIZATION AND FUTURE NEEDS FOR FIRE RETARDANT MATERIALS

by

A. J. McQuade
Clothing and Personnel Life Support Equipment Laboratory
U. S. Army Natick Laboratories
Natick, Mass.

The U. S. Army has a wide interest in nearly all aspects of fire, for it is the basis of many strategic and tactical munitions. Further, fire-type munitions are significant items within the arsenal of our potential enemies.

In view of the above, protection of our soldiers exposed to fire has been the subject of extensive work with the objective of including flame and thermal-protective materials in the soldier's combat uniform. Phosphorus, napalm, and the wide availability of gasoline form the basis for flame conditions, while the high-intensity radiant energy effects of nuclear weapons require that we reduce the hazard due to energy transfer through clothing layers to the skin.

In working on the problem of protective clothing for men subjected to such hazards, U. S. Army Natick Laboratories has carried out studies related to the chemical mechanism involved, the polymeric structure of materials, testing of materials in the laboratory by subjecting them to various types of thermal stress, clothing design and fabrication to enhance protection, exposure of full scale items to the actual munition of flame conditions, and finally, to prove the protection qualities of the items of materials, the use of fully clothed test animals exposed to these hazardous conditions.

In addition to the clothing worn by the combat soldier, the Army supply system includes other clothing items for specialty occupations. Among these special employees are firemen stationed at Army posts, camps, and stations. The items for firemen include: Coat, Fireman's OG 107; Trousers, Firemen; Gloves, Protective, Fire Fighters; and Hood, Fireman's Asbestos. In addition, there are fire protective clothing items for those working with explosive materials and in helicopters. Tentage has been the largest user of fire-retardant materials.

As a result, the Army is a large user and has been a purchaser of durable fire-retardant materials for the past 26 years. In addition, the Army has provided many millions of dollars for research and development in this field. However, the money spent for fire retardant battings has been very little. Perhaps the reason for this can be found in the term "durable" or in the term "fire retardant."

When the Army employs the terms "fire-resistant" or "fire-retardant" in its specification nomenclature, it is meant that the item or material is one that is self-extinguishing upon withdrawal of the applied flame. The flame to be applied is not the usual laboratory bunsen burner flame used in production testing, but a flame representing the one that will be encountered under actual use conditions. The conditions of fire met by the end item may or may not require resistance to afterglow.

When the term "durable" is applied, it is meant that the property of fire resistance, as I have described it, is maintained for the life of the item without any subsequent retreatment or restoration of the fire resistance properties. The reason for such a requirement is found when one examines the need for mobility in modern warfare. Today there are no field dry cleaning units as were available in World War II. Further, the number of field laundry units has been decreased. As a result, neither the manpower nor the equipment are available. Thus to achieve durable fire resistance, one must permanently attach fire-retardant treatments to the fibers or incorporate such chemicals within the fiber.

Tentage and paulins for the Army represent the first large-scale use of durable flame retardants. These were procured in large quantities during wartime. For example, 44 million square yards were delivered during 1 month of World War II—March 1945. The Korean War saw the attainment of 6 million square yards for several months. The present events in Southeast Asia only reached a peak production of 4 million yards due to the availability of a large stock purchased at the end of the Korean conflict. Replacement quantities are the only requirement at the present time, and during the calendar year 1968, this amounted to approximately 5 million yards.

For tentage, which one desires to man-pack or to be of much lower weight than can be produced using the fire-retardant materials for cotton duck, a two-bath application of a titanium and antimony oxide was available up to 1954. During the Korean War, a production rate of 2 million yards per month was realized with this type of treatment. This type of treatment is of very limited availability in the United States and England. Thus textile finishers have turned to cellulose reactive treatments based on THPC. For lightweight tentage fabric liners, where direct exposure to sunlight is avoided, several million yards were purchased in 1967 to replace depleted stocks. However, the conditions of Vietnam have not required large quantities and I can see no large requirements. A cotton sateen fabric used as the outer covering of lightweight tentage required by several Air Mobile Divisions has been the subject of several procurements; because of its replacement rate, it is expected that need for this type of fabric will continue. For this fabric, involving exposure to weather and sunlight, the THPC finish is modified by the addition of vinyl chloride and antimony oxide. Recent exposure tests have indicated that consideration should be given to its replacement with a modacrylic fiber fabric.

It is in the field of clothing that the utilization of fire retardant materials is growing. In 1968 the military services purchased or treated approximately 400,000 garments. For those of us who struggled about 15 years ago to produce an experimental quantity of 50 fire retardant uniforms for testing to determine what the problems might be, this represents considerable achievement. Explosive handlers' coveralls, firemen's clothing, and coveralls for aviators and ground crewmen represent the major outlets for such clothing. Under development are two new items for tankers' and aviators' winter clothing. Whether these will have complete testing and be ready for procurement in 1969 is unknown at this time.

The foregoing review completes what I can see as the Army's requirements for flame retardant materials for standard, or about to be standard items. However, it does not cover items for which the Army has stated there is a need for flame resistance and to which research is directed.

First, there is the clothing worn by the combat soldier, and perhaps his fatigue clothing worn for normal work in the continental United States. However, special items for this purpose are not desired. Fire and flame protection must be an integral part of his uniform and not have a deleterious effect on its functional properties or interfere with the soldier's performance. The only step in this direction which has been adopted is the use of cotton-nylon fiber blends in the outer layer of cold weather combat uniforms in order to inhibit their ignition when exposed to the thermal effects of nuclear weapons. This type of fabric is not effective when exposed to all types of flame-type weapons.

The insulating liners worn within cold climate clothing are a part of the soldier's uniform which need the property of fire resistance. In addition, there is the soldier's load carrying equipment as well as the items he may carry in this equipment. All should be fire resistant. The problem is not solely one of applying additive chemicals, but of incorporating these chemicals either in or on the fibers in such a way that durability of the fire resistance properties is obtained without sacrificing functional performance. Further, there can be no significant increase in weight.

In additon to the combat soldier's needs, fire resistance has been considered on several occasions for the soldier's bedding, be it in the hospital or the barracks. This would involve the mattresses, blankets, sheets, and pillows.

From what I have presented, it is apparent that the military, particularly the Army, has many needs for fire retardant materials. Some of these are satisfied with existing materials, others are partially satisfied, and there are many that are yet to be worked on. I hope from your meeting today there will be developed new products which will aid in meeting the Army's needs.

POTENTIAL FOR COTTON BATTING PRODUCTS

by

G. S. Buck, Jr.
National Cotton Council of America
Memphis, Tenn.

About 600 million pounds of cotton batting are used each year in bedding, automotive, and furniture uses. The total of all materials used as stuffing in these outlets is around 900 million pounds, so that the potential for cotton batting products may be assumed to be around 50 percent larger than present consumption.

The outlook for cotton batting consumption could, of course, be very much smaller than the 600 million pounds now being used annually. Factors that might bring about a downward trend include improvements in competitive products, changes in the price relationships between cotton batting and competitive materials, significant disparities in the merchandising effort for cotton batting and competitive materials, and new performance requirements for batting products. At this time it appears likely that fire resistance may be one such new requirement.

The potentials for cotton batting products, in the relatively near future, may depend heavily on the extent to which cotton batting can be made available with fire resistant qualities. This is the direct consequence of the amended Flammable Fabrics Act.

We all know that the present consumer demand for fire resistant properties in batting products is almost nil. Consumers will not willingly pay any significant premium to have the cushioning in their mattresses, furniture, or automobiles made fire-resistant. Not one in 10,000 units of these products ever catches fire; even when this happens, the likelihood of injury or fatality resulting is more remote by perhaps two orders of magnitude.

The economics of safety, in this case, require a special viewpoint to be acceptable. Depending on the cost of adding fire resistant properties to bedding, upholstered furniture, and automobile cushions, consumers may be required to pay up to a million dollars for the prevention of each accident involving batting ignition. I am not going to argue for a minute that such an investment should not be made. Certainly the victim of this type of accident would consider it worthwhile.

The fact we must all face is that along with the economics of safety we are dealing today with the politics of safety. The politics of safety call for almost any measure that will protect the public from accident and injury, no matter what the cost—as long as these measures do not interfere with the convenience of the public. I must add this exception because smoking, and primarily cigarette smoking, not only contributes directly to far more deaths than the combustion of fibrous products, but is also directly or indirectly responsible for almost half of all burning injuries involving textiles and probably 90 percent of all those that involve the combustion of batting products. The peculiar politics of safety, however, prevent our taking any direct action on this primary cause of these fires—cigarette smoking.

I think that what I have been saying about the economics of safety and the politics of safety are matters which have been in the minds of many batting manufacturers and users. The plain fact is, however, that there is not very much we can do about this situation. At one time we used to have a saying: “Don’t play with fire or you may get burned.” Today things have changed so radically that industry is called upon to make it impossible for anyone who plays with fire to be burned.

Given time, we can accomplish this goal. We can make completely safe fire-resistant cotton batting. It is quite likely that in doing this we will also make it easier to use complementary finishes which add resilience, moldability, mildew resistance, bacterial resistance, and other desirable properties.

While I have referred to aspects of new flammability and fire resistance regulations that, from an economic standpoint, may seem unreasonable, I am sure that every manufacturer of bedding, furniture, and automobile cushioning wants to do every-thing he can to prevent any injury or death. The only thing that troubles him is the tens or hundreds of thousands of units which must be protected at increased cost for each instance in which that protection is needed.

But we are living in a time in which we will be called upon to provide all the consumer protection which is technically feasible. If we do not do this with cotton batting, competitive products will be made available that do provide this type of safety. In my opinion we can provide fire resistance in cotton batting and at the same time make a superior product and even increase the potentials for cotton batting. We have a bigger opportunity in moving forward with the times than we have in resisting new regulations and in trying to hold back.

The attendance at this special Flame Retardant Cotton Batting Workshop is a practical demonstration that the cotton batting industry does, in fact, intend to move ahead. It is especially gratifying that the Southern Regional Research Laboratory (SRRL) is participating in the sponsorship of this meeting. SRRL scientists have already demonstrated what they can do with cotton batting in the development of the resilient Cotton Flote.

It is also gratifying to have the active participation in this effort of many chemical manufacturing firms. Their contributions will be very important to our ultimate success in these efforts. We would simply caution these firms against claiming, in their zeal to sell chemicals, prematurely to have solved all the problems of flame-retardant batting. (And here I am setting an example for chemical manufacturers by being very conservative.)

All in all, the new problems we face, and the new requirements we may be called upon to meet, really amount to an opportunity for cotton batting. They constitute a way to build a better and a safer product for consumers, and at the same time, to build a more viable and more profitable cotton batting industry.

FIRST SESSION: J. M. Bing, Royal Manufacturing Co., Charlotte, N.C. Chairman

THE FLAMMABLE FABRICS ACT

by

S. L. Sutcliffe
Senate Commerce Committee
Washington, D. C.

In 1967 Congress passed the Flammable Fabric Act Amendments. It was their conviction that burns from the ignition of clothing and other household fabrics constituted a very serious health problem. Deaths from fire rank third—behind traffic and falls—as the chief cause of accidental deaths and injuries in this country. The hearings on the Flammable Fabrics Act Amendments disclosed that the death rate from burns is twice as great as the number of cases—not deaths, but cases—of polio that occurred in the prevaccine era. Statistics on the death rate from fires, however, do not begin to reveal the magnitude of the problem. Many of the victims of fire do not die, but suffer second- and third-degree burns involving 10, 20, and even 30 percent of their bodies. They suffer anguish that can scarcely be described, let alone endured. If they survive, many of them are permanently disabled or disfigured. Often the survival is achieved at the cost of medical and surgical treatment reckoned not in hundreds but in many thousands of dollars—and not in days or weeks but in many months of hospitalization. The highest proportion of people burned are those least able to help themselves—the aged, the disabled, the poor, and the young.

The 1967 Amendments passed by Congress transformed the limited Flammable Fabrics Act directed at explosive clothing into a comprehensive fire safety law for all household and personal fabrics. This transformation was accomplished: (1) By giving the Secretary of Commerce the authority to promulgate standards or other appropriate regulations whenever he finds that such action is needed to protect the public interest; (2) by extending the scope of the original act by including all "articles of wearing apparel" and "interior furnishings"; (3) by directing the Secretary of Health, Education, and Welfare to conduct a comprehensive and continuing investigation of the deaths, injuries, and economic losses resulting from accidental burns; and (4) by authorizing the Secretary of Commerce to conduct research in the flammability of fabrics and related subjects.

Under this legislative scheme all "interior furnishings" are within the scope of the Act. "Interior furnishings" are defined as "any type of furnishing made in whole or in part of fabric or related material and intended for use, or which may reasonably be expected to be used, in homes, offices, or other places of assembly or accommodation." Bedding and related products, therefore, are clearly within the scope of the Flammable Fabrics Act as amended. But that does not mean that these products will automatically be affected by the amendments.

Only after investigation and research by the U. S. Department of Commerce, the U. S. Department of Health, Education, and Welfare, or private organizations and after a determination by the Secretary of Commerce that a particular product presents an "unreasonable risk" of death, personal injury, or significant property damage will that product be subjected to flammability standards or labelling requirements. In other words, the legislation is designed in such a way as to affect only those products that have been shown to create an unreasonable risk to the consuming public.

An "unreasonable risk" may be defined as one where the probability of an injury and the seriousness of that anticipated injury, when considered together, outweigh the burden—to manufacturer and consumer—of eliminating or reducing the injury. Before any determination of "unreasonableness" can be made, one must know the probability factor, the seriousness factor, and the burden factor. If the probability of injury is high, and that injury is likely to be severe, and the burden of eliminating the injury is not great, then the Secretary of Commerce will find that the product presents an "unreasonable risk", and he will promulgate standards to eliminate such risk.

Of course there may be differences of opinion as to what is "unreasonable." The rule-making activities of the Secretary of Commerce are subject to the Administrative Procedures Act. Therefore, proposed rules must be published in the Federal Register and there is opportunity for comment by interested parties before final adoption. Any aggrieved party can seek judicial review of the Secretary of Commerce's determination. In addition, the Act provides that a National Advisory Committee representing manufacturers, distributors, and the consuming public will be consulted before any standards are passed. Given the legal guarantees and the more informal but perhaps more significant consultation with the National Advisory Board or workshops such as these, no harsh arbitrary rules should be fostered. Under the Flammable Fabrics Act as amended, the interests of all parties will be protected while the health of the American people will be improved.

Having explained the Flammable Fabrics Act in general terms, it might be useful to examine how the cotton batting industry might be affected by the Act. Statistics from a recent survey indicate that the articles from which the greatest number of deaths and injuries result are items of bedding and upholstered furniture. If further investigation verifies the results of this survey, then bedding and upholstered furniture could be considered high risk items. Let us assume that the burden of reducing the fire hazard associated with these items is not great enough to foreclose taking steps to reduce the hazard. Under these circumstances the Secretary of Commerce would propose a flammability standard for these high risk products. After receiving comments on the proposed standards and consulting with the National Advisory Committee, the standards would be finalized.

Suppose a standard for mattresses states that all mattresses must be able to withstand a temperature of *X* degrees for *Y* minutes. How would such a standard influence the cotton batting industry? On the one hand it may have no effect on cotton batting if a ticking is available which would meet the standard and if mattress manufacturers were willing to use such ticking. On the other hand a flame retardant ticking may not of itself be sufficiently retardant, or mattress manufacturers for a variety of reasons may prefer to use a combination of flame-retardant ticking and flame-retardant batting. In any event the industry would have a certain amount of flexibility in deciding how best to satisfy a flammability standard designed to eliminate at least one type of bed-related accident.

CONGRESS

SECRETARY OF COMMERCE

Fabric Flammability Section
Product Evaluation Division
National Bureau of Standards

1. Data accumulation . . . .) U.S. Dept. of Health
2. Testing methods . . . .) Education, and Welfare
3. Independent investigation

STANDARDS

1. Publication; opportunity for hearings
2. National Advisory Committee
    1. Manufacturers
    2. Distributors
    3. Consumer representatives

Injury Control
Program

Burn Study Centers

National Burn
Information Exchange,
Ann Arbor, Mich.

Consumers Union

Information Council on
Fabric Flammability

U.S. Dept. of Agriculture
flame-retardant research

Other
fire marshalls
government agencies

IMPLEMENTATION OF THE FLAMMABLE FABRICS ACT

by

J. V. Ryan
National Bureau of Standards
Washington, D.C.

INTRODUCTION

The original Flammable Fabrics Act was enacted in 1953 in response to burn deaths and injuries of several persons, caused by the notorious "torch sweaters" and play suits. It was limited to wearing apparel, and even a few items of that were exempted. The 1967 amendments to the Act enlarged its scope to include all wearing apparel and all interior furnishings. They authorized the Secretary of Commerce to set standards for items that represented unreasonable risk to the public, subject to certain procedural safeguards. They authorized research and investigations, test method development, and called for a National Advisory Committee for the Flammable Fabrics Act. You have heard Lynn Sutcliffe describe the amendments in detail, and indicate the implications in the context of your interests in cotton batting.

INTERPRETATION

The interpretation put on the provisions of the Act will determine the directions in which National Bureau of Standards (NBS) goes and the philosophy that guides us in our choices of areas of interest and of priorities.

The Act defines interior furnishings as any type of furnishing made in whole or in part of fabric or related material and intended for use, or which may reasonably be expected to be used, in homes, offices, or other places of accommodation. In my mind, there is no question that bedding, and mattresses are interior furnishings in the meaning of the Act. I believe that the legal minds of the Department of Commerce hold the same opinion.

The Act speaks of unreasonable risk to the public, of death, injury, or economic loss resulting from the ignition of items subject to the Act. No definition is given for unreasonable risk, or what constitutes it. Certainly, it was not the intent of the Congress to attempt to eliminate all risk, but many of us face risks every day without knowing of them, or without the means of doing much about them. These are the unreasonable risks that were of concern to the Congress and are to us. This may be an oversimplification, but I, too, wish to retain my freedom to exercise judgment as the occasion demands.

The Act refers to fabrics, products, and related materials. While it may give equal emphasis to each, it seems that the appropriate approach is to products, rather than to materials, in the first instance. Certainly it is possible (many examples could be cited) for a material to be safe for one use but not for another. Should such a material be ruled off the market? I doubt it! With a little ingenuity, any of us could imagine an unsafe use for almost any material. Rather, I believe the correct approach is to seek out the products that represent unreasonable risk to the public and set reasonable standards of performance which, when met, will eliminate or reduce the risk. This will accord American industry full freedom to modify existing materials or substitute other materials in manufacturing products. Let the role of government under the Flammable Fabrics Act be that of determining the level of protection to be assured to the public; let industry and the play of the marketplace determine what materials and combinations of materials, what designs and constructions, will be used to achieve the required performance.

IMPLEMENTATION

Before trying to apply the above general interpretation to the specifics of cotton batting, let me discuss what is being done and what is some of our thinking about what we hope to do.

The investigations of deaths and injuries required by the Act have been going on for some time. Over 2 under specific conditions. Rather than taking off from there we are considering what the hazards are, what needs to be measured, and how these things can years ago, the National Bureau of Standards and the U. S. Public Health Service (USPHS) entered into an agreement whereby the investigating teams (then only one, in Denver) of USPHS send us reports of burn cases involving wearing apparel and, whenever possible, send us the garments involved. We make an examination to identify the fiber content of the garment and conduct the standard flammability test that is the basis of the present regulations. We provide reports of the results to USPHS. So both agencies have been building up a pool of data. Their efforts were concentrated on burn cases (the teams originally were set up to investigate all kinds of accidents) when passage of the legislation appeared certain.

The National Bureau of Standards established the Fabric Flammability Section, within a few weeks after the passage of the amendments, to carry out the responsibilities for research and investigation assigned to the Secretary of Commerce. Our staff is small and most of our time has been used for analysis of the data from Public Health and for administrative matters related to starting a new program. We were involved in the preparation of a program plan, a budget, appropriation request, statements of the Department procedures for developing flammability standards, and identification of candidates for the National Advisory Committee. Until recently, the technical staff consisted of three scientists, only two of whom had been involved in fabric flammability studies for any appreciable time. My background had been in fire, without fabric specialization, and legislation. We three were joined by a secretary to make a Section that served to identify and consolidate the on-going effort, but did not represent an increase in the level of effort. A decision was made by the Bureau of the Budget not to send a request to the Congress for supplemental appropriation for the remainder of the fiscal year that ended last June 30. The appropriation for the current fiscal year was enacted just before the Congress recessed for the political conventions, but allocations of funds could not be made until all appropriations were enacted. Consequently, the first new funding for the program became available only recently. We have taken on one new man and hope to add three or four more in the next few months.

The Department of Commerce published proposed procedures for rule-making, received comments thereon, and published the final procedures. They appeared in the Federal Register of October 1, 1968. They provide several steps to the development of standards. These are: a somewhat general finding that a new or amended standard may be needed; a minimum period of 30 days for comment on the finding and/or on the terms and substance of a flammability standard if it is decided one is needed; promulgation of a proposed standard; another minimum 30 day period for comment; the opportunity for requesting informal hearings; submission of the proposed standard to the National Advisory Committee for their comment; a meeting of the Committee to discuss it; individual written comments from each member to the Secretary; and, only then, publication of the standard with the final finding that the specific standard is needed. As you see, interested parties are afforded repeated opportunities to participate by submitting comments and supporting information or data. Following final publication, an adversely affected party may petition the courts for judicial review of the proceedings. The procedures will require several months; a reasonable least time would be 6 to 9 months.

The Act calls for a National Advisory Committee, of not less than nine, to be fairly representative of manufacturers, distributors, and the consuming public. In addition to the problem of selecting the best people from the several dozens of names suggested, or identified internally, there were problems as to the legal interpretation of "fairly representative." There were opinions that it could be satisfied only by equal representation; opinions that the consumer could be safeguarded only if the representatives of the consuming public outnumbered the combined representation of manufacturers and distributors; and opinions between these. There were other questions, such as could persons be appointed to represent general interests? We are all consumers as individuals, but a member of the fire services, for example, would have an interest, as a result of his work and beyond that of a consumer of fabrics and related materials. Reasonable positions were reached in connection with all these questions. The Committee will represent a well-balanced group of interested individuals capable of looking at the numerous facets of regulations from many points of view. Not all are expert in, nor even familiar with, textiles or flammability, but I belive you will find each is capable of an intelligent approach.

Our research to date is mostly in the thinking and planning phase. Although the research is not confined, nor even largely devoted, to cotton batting, I will discuss our thinking in its application to cotton batting.

We are going to what are often called first principles. The present test method measures flame spread be measured with meaning related to the hazards to the public. The speed of burning for a fabric may be of critical importance, if used in a garment. Quick removal of the garment can prevent or minimize injury. However, the amount of heat that is liberated, and transferred to the skin, will determine the severity of the resulting burn. If the fabric or material is used in furniture or bedding, the potential victim may be in a different part of the building than that in which the item burns. Then the heat may be of lesser importance. Cotton batting's most important use are in furniture and bedding, as far as the Flammable Fabrics Act's scope is concerned.

Fire victims often are found in relaxed positions, sometimes not even in rooms directly involved in the fire. The implication is that they never knew what hit them, or even that they were being hit. In many cases, it is obvious that flames did not reach the victim at all. But even in those cases where the corpse is charred, it is hard to imagine that the flames got there first. Had that been the case, you would expect that there would be some indication that the victim tried to escape or to fight the fire. All the evidence suggests that fumes were the prime culprit, in that they either killed the victim, or deadened his senses against the attack of a less insidious phase of the fire.

Smoke is another product of a smoldering fire that may be either a direct killer or an indirect, contributing factor. If you or I awaken to find our respective homes filled with smoke, we probably stand a good chance of finding our way out of those familiar surroundings without being able to see. Assume unfamiliar surroundings such as a hotel, vacation retreat, or another's home that we are visiting, and the results may be that we are trapped while flames or searing gases find us out. Lacrimatory or other blinding fumes or vapors could have the same deadly effects as smoke. Of course, ingesting enough smoke will produce death without any other agent.

Our approach will be that of identifying the conditions of use for a particular class of products, and the hazards that logically are connected with that use. For most of the products in which cotton batting is used, these could include ease of ignition, very rapid and intense burning, production of smoke, and production of toxic, blinding, or anesthetizing fumes or gases.

A question that was asked in a meeting several weeks ago had to do with the need to make cotton batting flame resistant since it is ordinarily used beneath a decorative or wearing material. There is no pat answer to that question, except the general answer that any regulations are expected to set performance requirements for the assembly, the end product. *If* the characteristics of the covering material are such that the cotton batting beneath *cannot* be brought to flaming combustion, or smoldering combustion, or to thermal decomposition (so that no hazard is connected with the characteristics of the cotton batting), then it need not have any particular fire characteristics. That strikes me as a big "*if*". For example, a leading tester of consumer products recently evaluated a mattress manufactured with a flame retardant treated ticking. When a cigarette was laid on a combination of the ticking and batting, the ticking charred but did not burn or glow, so the batting could not be ignited by the ticking. However, the charred ticking was so brittle that it broke letting the glowing cigarette into direct contact with the batting. The fire retardant ticking was not a direct hazard, but it did not eliminate the possibility that the batting beneath could become involved. Actually, the mattress marketed had both ticking and batting treated, but the testing organization was checking on the possible combination of treated ticking and untreated batting. For this particular ticking, at least, it would be of doubtful effectiveness and of doubtful safety.

As far as any possible regulations under the Flammable Fabrics Act might go, the overall performance of the assembly will be the determining feature. The performance of any constituent material, or combination of materials, will be of concern only to the extent that it contributes to or predicts the total performance. This does not necessarily imply that all regulations will be based on methods requiring the test of full scale, end use products. As I mentioned previously, one of our concerns is to determine what needs to be measured, and another is to devise convenient means for measuring those things found to be significant. At the outset, this may require the burning of complete beds in suitably sized and instrumented test rooms, in order to find out more than is now known about the growth of a bedding fire, the temperatures reached, and the concentrations and kinds of products of combustion. Once we get reliable and representative values, I hope we can devise methods that will permit measurement of relatively small specimens. It would be unfortunate to have to burn full mattresses (or full carpets, dresses, sofas, etc.) in order to determine their compliance with applicable regulations. Full scale tests increase the difficulty of controlling all the possible variables. They also increase the cost of test specimens, test apparatus, and test operation.

DISCUSSION

Question: Would you have statistics on how many incidents of death or injury result from mattress or furniture fires? I would like to have this figure as a percentage of households or unit sales of mattresses or furniture.

Mr. Ryan: As was indicated in my presentation and Mr. McGuire's talk at the luncheon, there are no reliable statistics that can be quoted that would establish the hazard potential. It is hoped that the survey now being made by the Department of Health, Education, and Welfare will shed some light on the matter. At present each state, city, county, or other agency reports deaths and injuries from fires in their own way and there is little consistency from one to the other. Frequently, autopsies made after fires are cursory and little or no effort is made to distinguish between deaths from actual burns and those by asphyxiation. As a consequence, the figures that are available and frequently quoted contain much questionable data.

Question: Would the use of an approved flame resistant mattress pad eliminate the need for a flame resistant mattress?

Mr. Ryan: Will you let the Government come into your bedroom to make sure you are using that mattress pad? I think the answer obviously is, no.

Question: Will the Flammable Fabrics Act make it more difficult to obtain approval for marketing a new product? That is, will the standards for a new product be more severe than for existing products?

Mr. Ryan: I am not sure what is meant by a *new* versus *existing* product. Let's talk again about mattresses. There may be requirements for mattresses. If somebody comes out with a new construction for mattresses—a totally new construction—presumably it will have to meet the same requirements as existing mattresses if requirements are set for mattresses.

Question: Will the Commerce Department consider the factor of burden in relation to unreasonable risk—the possible undue advantage of one type of manufacturer to the detriment of another should the fire resistance standards be adopted? I am thinking of the possible advantages of foam mattresses over the cotton-filled type because of price or lack of equipment in the past in the cotton industry.

Mr. Ryan: I don't wish to seem snide, but the answer is, yes, these sort of things will be considered.

Question: How will industry be notified and what lead time will be given when the new fire retardant requirements are proposed?

Mr. Ryan: The lead time is the axis that the standards go into effect 12 months after they are officially promulgated. How will you know about it? Industry will be represented on the National Advisory Committee. These things will all be published in the Federal Register, which is sort of the official newspaper for the Federal Government. I am sure that a lot of you will be hearing through the Office of Textiles, whose function, as mentioned this morning, is industry contact.

Question: Can it be estimated in time when implementation of the Nonflammable Act will crystallize to the point where manufacturers will have to supply a nonflammable product—mattresses in particular?

Mr. Ryan: I think the answer is essentially the same one that I gave this morning. I agree with George Buck—that the chances are fairly good and that it will come sometime.

Question: Is there a specification covering noncombustibles in the aircraft of today and the SST of tomorrow? Or, is it also covered by the Flammable Fabrics Act of 1967? What part does the Federal Aviation Agency (FAA) play?

Mr. Ryan: It would not be covered by the Flammable Fabrics Act per se. It is covered by the authority of the Department of Transportation and particularly in this case by FAA which is part of the Department of Transportation. They do have specifications.

FLAME RETARDANT COTTON—STATE OF THE ART

by

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INTRODUCTION

President Johnson signed Public Law 90-189, an Act to amend the Flammable Fabrics Act of 1953, to provide increased protection for consumers against injurious flammable fabrics on December 14, 1967, in Washington, D. C. On that same day the first annual meeting of the Information Council on Fabric Flammability was held in New York City.

In an invited talk which I presented to the Information Council on the State of the Art of Flame-Retardant Fabrics at that time (8), I stated that practical achievement of the goals of the 1967 Flammable Fabrics Act Amendment will require the maximum possible communication and cooperation between and among the various industrial, governmental, educational, and professional organizations associated with the development of the improved flame-retardant fabrics and related materials which are needed. Accordingly, I am very pleased to participate in this workshop for the development of improved flame-retardant cotton products, which is well-attended by many leaders of all the required cooperative organizations.

The formation of the Information Council on Fabric Flammability was one result of a Conference on Burns and Flame-Retardant Fabrics which was also held in New York City in December 1966. At the 1966 conference, a spokesman for the American Textile Manufacturers Institute *(16)* reported that the chemical treatment of cellulosic fabrics or some unknown blend offered the best approach for the mass production of the improved flame—retardant fabrics, which are needed to provide adequate fire safety, without sacrificing other important properties that are required or desired by the consumers.

However, due largely to the increased cost and lack of comfort of durably flame-retardant fabrics available at that time, the U. S. textile industry representative reported that it was not then feasible to write specifications which could be approved by the textile industry for acceptable mass consumer apparel uses. Nevertheless, during the past 35 years, the U.S. Department of Agriculture, the U.S. Department of Commerce, and the U.S. Textile and Chemical Industries have continuously cooperated with U.S. military agencies and other scientific and technical organizations for the development, production, critical evaluation, and preparation of adequate performance test specifications for durable fire-resistant cotton textiles. Most, but not all, of the flame retardant fabrics so produced however, have been for military and industrial markets.

In the very limited time allotted for this talk, I plan to review briefly and identify the principal practical developments resulting from this past industry and government cooperation. Principal emphasis in my discussion will be given to the durable fire-resistant finishes for cotton which have survived commercially from the many attempts to develop and market fire-resistant cotton products and related, competitive fire-resistant materials during the past 35 years.

The durable fire-resistant chemical finishes which I will identify can be used to produce cotton fabrics which retain most of their fire resistance as measured by the vertical flame test of the American Association of Textile Chemists and Colorists and related Federal specifications, throughout their useful life. Less durable flame retardants are usually referred to a semidurable, partially durable, or nondurable. As their use in the development of flame-retardant cotton batting will be discussed by following speakers on today's program, I plan to only mention briefly the nondurable and semidurable flame-retardants.

Past Government-Industry cooperation for fire resistant cotton fabrics—About 35 years ago the then Bureau of Chemistry and Soils of USDA developed a laboratory method for imparting fire, water, and weather resistance to cotton canvas fabrics *(9)*. Although this method was not commercially or technically feasible, it stimulated and led to the development, in private industry, of the first practical and still most widely used *durable* fire-resistant finish for cotton. Fire resistance and other functional properties of this finish are largely retained throughout the useful life of the finished cotton fabric, which varies from 5 to 15 years of constant outdoor exposure. This finish has been referred to since its original development as the FWWMR, which stands for fire-, water-, weather-, and mildew-resistant *(5)*. Following critical evaluations by the National Bureau of Standards, the Underwriters' Laboratories, the U.S. Army Corps of Engineers, and the U.S. Quartermaster Corps from 1935 to 1939, a Federal performance specification for this fire resistant cotton product was agreed on by government and industry in 1939 *(7)*. Since then many hundreds of millions of yards of FWWMR treated cotton fabric have been produced under rigid specification requirements, principally for military but also for civilian markets for outdoor fabric use.

FWWMR production during World War II has been reported to involve the largest textile procurement in our government's history (*13*). Accordingly, with the possible exceptions of glass and asbestos fabrics, this type of finish accounts for the greatest amount of durable fire resistant fabric ever produced by industry. The great majority of this FWWMR production that has continued up to the present has met rigid Federal and related military specification test standards, for fire resistance and other numerically measurable functional properties.

Such standards, therefore, may provide helpful precedents and guides for the establishment of the new standards for various end uses which will be required for constructive implementation of the 1967 Flammable Fabrics Act. New standards required as competition and among various fire resistant products made of differing fibrous or plastic materials can only be kept on a fair, objective basis and be of realistic, honest benefit to the consumer, if the original performance specifications are fairly and equitably prepared and rigidly adhered to.

Since World War II, the Southern Regional Research Laboratory (SRRL) of USDA, one of the sponsors of this workshop, has developed improved fire-resistant processes for cotton apparel fabric which have become known as the THPC (*15*) and APO-THPC *(14)* finishes. These finishes are essentially organic compounds or compositions containing phosphorus and nitrogen. They were developed in cooperation with the U. S. Army Quartermaster, now the U.S. Army Natick Laboratories—represented here today by Allan McQuade.

Table 1 illustrates the typical amounts of phosphorus and nitrogen contained in fabric treated with the THPC and APO-THPC finishes. In addition, this table illustrates the following:

1. Typical amounts of antimony and chlorine contained in an FWWMR finished fabric. The essential fire resistance of the FWWMR finish is produced by a synergistic combination of antimony oxide and a chlorine-containing organic compound.
2. The partially durable flame retardant, urea phosphate, which is an organic compound containing phosphorus and nitrogen, in combination with antimony oxide and polyvinyl chloride. The percentage of antimony, chlorine, nitrogen, and phosphorus typically contained in fabric finished with the preceding composition are also listed.
3. The relative percentages of phosphorus and nitrogen contained in a cotton fabric treated with a typical THPC finish.
4. The relative percentages of phosphorus and nitrogen contained in fabric finished with melamine polyphosphate, a partially durable fire retardant.
5. The relative percentages of antimony, chlorine, and phosphorus contained in a finished apparel fabric finished with a composition containing antimony oxide, chlorinated compound, and THPC.
6. The relative percentages of phosphorus and nitrogen contained in fabric finished with a typical APO-THPC finish.
7. An example of a bromine containing organic compound, brominated allyl phosphate, in combination with THPC. Although bromine-containing organic compounds have not had significant commercial success as flame retardants in the past, they have been receiving increased attention in industrial research organizations in recent years.

Current commercially used durable fire resistant finishes for cotton—The Chairman of the American Association of Textile Chemists and Colorists' Committee on Fire Resistance Test Methods, Dr.

Table 1.—Typical THPC and APO-THPC finishes

Flame retardant	Dry and cure conditions	Contents of finished fabric
Antimony oxide Chlorinate compound	Dry only	7 pct. antomony 3.5 pct. chlorine
Urea phosphate modified with Sb_2O_3 vinylchloride	Cure	3.5 pct. antimony 1.6 pct. chlorine 2.4 pct. nitrogen 2.4 pct. phosphorus
THPC	Dry cure 4.5 min. at 285° F.	1.7 pct. phosphorus 5.5 pct. nitrogen
Melamine polyphosphate	Dry cure 3 to 5 min. at 300° F.	1.5 pct. phosphorus 7.9 pct. nitrogen
Antimony oxide Chlorinated compound modified with THPC	Dry and cure 2 min. at 320°	3.3 pct. Sb 1.2 pct. phosphorus 2.1 pct. chlorine
APO-THPC	Dry cure 5 min. at 300°	2.6 pct. phosphorus 1.6 pct. nitrogen
BAP-THPC	Dry cure 5 min. at 284°	1.6 pct. phosphorus

Bruce LeBlanc, has permitted us to reproduce tables from his current article in the October 1968 issue *Textile Industries* on "Guide to Flame Retardants" *(10)*. These tables provide us with a brief, but more detailed identification of the types of durable flame retardant finishes which have survived commercially, from the many attempts to develop fire- or flame-resistant textile products during the past 35 years.

Table 2.—Typical FWWMR finish

Component	Percentage
40 percent chlorinated paraffin	13
70 percent chlorinated paraffin	7
Antimony oxide	7
Binder	7
Pigment	9
Calcium carbonate	4
Aluminum stearate	1
Mildew resistance agent	1
Organic solvent	51

Cotton duck is padded to 100 percent wet pickup, and dried

Table 2 illustrates a typical FWWMR finish formulation. This type of finish has in the past 35 years accounted for the greatest amount of durably fire-resistant textile material of any kind ever chemically produced by industry. The fire resistance property of this multifunctional finish results from a synergistic combination of antimony oxide and a chlorine containing organic compound—in this case chlorinated paraffin. The FWWMR finish uses organic solvents as vehicles for dispersion and application of the essential multifunctional components to the treated fabric. It therefore provides a practical example of the effective use of organic solvents in textile finishing processes, which has recently become a subject of increasing potential importance in textile finishing research and development.

Recent production of FWWMR finished cotton fabric has been estimated by Dr. LeBlanc to be about 50 million yards per year—mostly, but not entirely for military fabrics for outdoor, nonapparel uses.

Table 3 illustrates the type of finish which has accounted for the largest amount of durable fire-resistant cotton fabric for apparel uses that has been produced since World War II. This finish combines the synergistic antimony oxide, chlorine-containing organic compound fire-retardant system first developed for FWWMR in industry, with the phosphorus- and nitrogen-containing organic compound system THPC, first developed for durable fire-resistant cotton apparel fabrics at the Southern Regional Laboratory of USDA. The THPC imparts afterglow resistance and supplements the fire resistance of the antimony oxide-PVC combination.

Table 3.—Typical THPC, Sb_2O_3, and PVC finish

Component	Percentage[1]
Trimethylol melamine	8.4
Triethanol amine	4.2
Tetrakis (hydroxymethyl) phosphonium chloride	11.2
Urea	4.2
Polyvinyl chloride	11.0
Antimony oxide	11.0
Wetting agent	0.5
TOTAL SOLIDS	50.0

[1] Percentage of solids in liquor.

Padded to 70 percent wet pickup on an 8-oz fabric, such as a twill or sateen, dried, cured about 2 min. at 325° F, washed in hot perborate and soda ash, and rinsed. Fabric is then top-softened and dried.

Fabric will not propagate flame even after more than 25 commercial launderings.

Recent production of cotton fabric treated as in table 3 has been estimated by Dr. LeBlanc at about 20 million yards annually, mostly for military and industrial clothing markets. Cotton fabric properly finished by formulations such as table 3 will pass the vertical flame test of the AATCC after more than 25 commercial launderings.

It should be pointed out that the components of the table 3 finish are applied from dispersions in water instead of dispersions in organic solvents as in the FWWMR composition listed in table 2. In the past, most textile finishing firms have preferred not to use organic solvents, because of possible fire, health, and explosion hazards, or because of the additional costs of solvent recovery systems.

Table 4 describes a typical APO-Thiourea finish, which is an example of an organic flame-retardant composition containing essentially phosphorus, nitrogen, and sulfur. Although about 1 million yards of cotton apparel fabric were reported to have been successfully treated with this finish for military specification procurement in recent years, the principal past chemical producer of APO (Tris 1-aziridinyl phosphine oxide) has withdrawn it from commercial markets within the past year.

However, this finish has been reported by Dr. LeBlanc to account for the third largest commercial production of durable fire resistant cotton fabric produced in accordance with rigid specification requirements in recent years. The APO-Thiourea finish is also applied from aqueous dispersions. Cotton fabric properly finished by formulations such as in table 4 will pass the AATCC vertical flame test after 25 commercial launderings.

Table 4.—Typical APO-Thiourea finish

Component	Percentage[1]
Tris (1-aziridinyl) phosphine oxide	21.5
Thiourea	11.5
Wetting agent	0.1
Softener	1.0
TOTAL SOLIDS	34.0

[1] Percentage solids in liquor.

Padded to 70 percent wet pickup on an 8-oz fabric, such as twill or sateen, dried, cured for 2 min. at about 325° F, washed in a little wetting agent, rinsed, and dried.

Fabric will not propagate flame even after more than 25 commercial launderings.

Table 5.—Typical straight THPC finish

Component	Percentage[1]
Tetrakis (hydroxymethyl) phosphonium chloride	32.0
Caustic soda	3.5
Trimethylol melamine	8.2
Urea	6.5
Softener	1.0
Wetting agent	0.1
TOTAL SOLIDS	51.0

[1] Percentage solids in liquor.

Padded to 70 percent wet pickup on an 8-oz fabric, such as twill or sateen, dried, cured 1 min. at 325 ° F. Wet cured at 140 ° in solution of ammonium sulfate and soda ash (ammonia cure). Washed in perborate and soda ash, rinsed, and dried.

Fabric will not propagate a flame after more than 25 commercial launderings.

Table 5 describes a typical straight THPC finish which identifies the fourth largest market volume durable flame-retardant finish for cotton fabrics. Although it produces fabric with a better hand than the finish described in table 3, it is also more expensive and therefore was reported by Dr. LeBlanc to have achieved very limited market acceptance. However, cotton fabric properly finished with the composition listed in table 5 will pass the AATCC vertical flame test after more than 25 commercial launderings. This finish is also applied to the fabric from aqueous media.

Current trends and future possibilities.—Most of the recent industrial production of the previously identified durable fire-resistant finishes for cotton fabrics has been for military uses.

Table 6 provides a view of the past markets for the various classes of flame-retardant cotton fabric finished in the less warlike year of 1963. According to estimates of the Market Research Division of the National Cotton Council of America, of a total production of 38 million yards of all types that year, the FWWMR accounted for 16 million, other durable and semidurable types 13 million and the nondurable type 9 million linear yards.

Table 6.—Estimated amount of flame-retardant fabric finished in 1963. (Most recent figures)

Type finish	Linear yards	Pounds
	Million	*Million*
FWWMR	16	10
Other durable, semidurable	13	5
Nondurable	9	4
Total	38	19

Accordingly, it is evident that fire- or flame-resistant fabrics have achieved only very limited consumer demand in the past, as the total U.S. production of all broadwoven fabrics has amounted to about 17 billion square yards annually in recent years, cotton fabrics accounting for about two-thirds of the total square yardage (*2*).

However, government and industry research organizations have re-intensified their past efforts to develop improved flame-retardant textiles and related materials since the passage of the 1967 Flammable Fabrics Act, in anticipation of potential, mandatory Federal requirement in large consumer textile markets.

Accordingly, reports were made at the recent 17th Annual Chemical Finishing Conference of the National Cotton Council on research advances in industry and government on durable flame-retardant systems based on organic compounds containing phosphorus and nitrogen.

One of these was a report of a more economical durable flame-retardant finish for cotton that has been developed at the Southern Regional Research Laboratory of USDA under the direction of Wilson Reeves, Chief of the Cotton Finishes Laboratory. The finish is based on the THPOH-NH_3 cure system which is derived from the THPC system originally developed at SRRL by Mr. Reeves and Dr. John Guthrie. In addition to lower cost, improved SRRL flame retardant development product (*4, 6*) has been reported to have a better "hand" than the durable flame retardants for cotton apparel fabric that I have previously identified.

Also reported on at the recent Chemical Finishing Conference is another phosphorus-nitrogen containing durable flame retardant system which has been commercially used on cotton fabrics in the past year. Informal reports from reliable textile research leaders indicate that this phosphonate finish of a Swiss firm does produce a product with superior hand and has accounted for a commercial production of more than one-quarter of a million yards of finished all-cotton fabric so far in 1968 (*1*).

An additional pilot method for imparting durable fire resistance to cotton fabircs with organic compounds containing phosphorus and nitrogen was also dexcribed. This novel method utilizes high energy radiation to produce durable flame-retardant cotton textiles (*11*).

Such durable fire-resistant all-cotton products form a carbonaceous, somewhat heat insulating char when decomposed by flames. This is shown in figure 1, which also illustrates the superior consumer protection provided by such all cotton fire resistant fabrics.

Since the beginning of the 20th century, it has been recognized that it would be particularly desirable to impart such fire protection to cotton flannels, which have been continuously used since then for children's nightwear, largely because of their superior comfort and other desirable properties.

In their Christmas Catalog for 1968, page 41, Sears, Roebuck, and Company is offering girls' cotton flannel robes, gowns, and pajamas treated with Sears' unique and otherwise unidentified finish, which I understand will pass the AATCC vertical flame test after at least 50 washings. Figure 2 illustrates this most recent national market exploration of consumer demand and acceptance of durable fire-resistant cotton garments.

The National Cotton Council and the National Cotton Batting Institute have reported this year on their excellent demonstration of the superior fire safety of a flame retardant cotton mattress at the 40th Annual Fire Department Instructors Conferences in Memphis last March (*12*). Figure 3 illustrates the breakthrough in mattress safety which can be made available to consumers by imparting fire resistance to cotton batting.

The following speakers on this workshop program will provide you with much additional information for the present and future development and production of flame retardant Cotton Flote as well as flame retardant cotton batting.

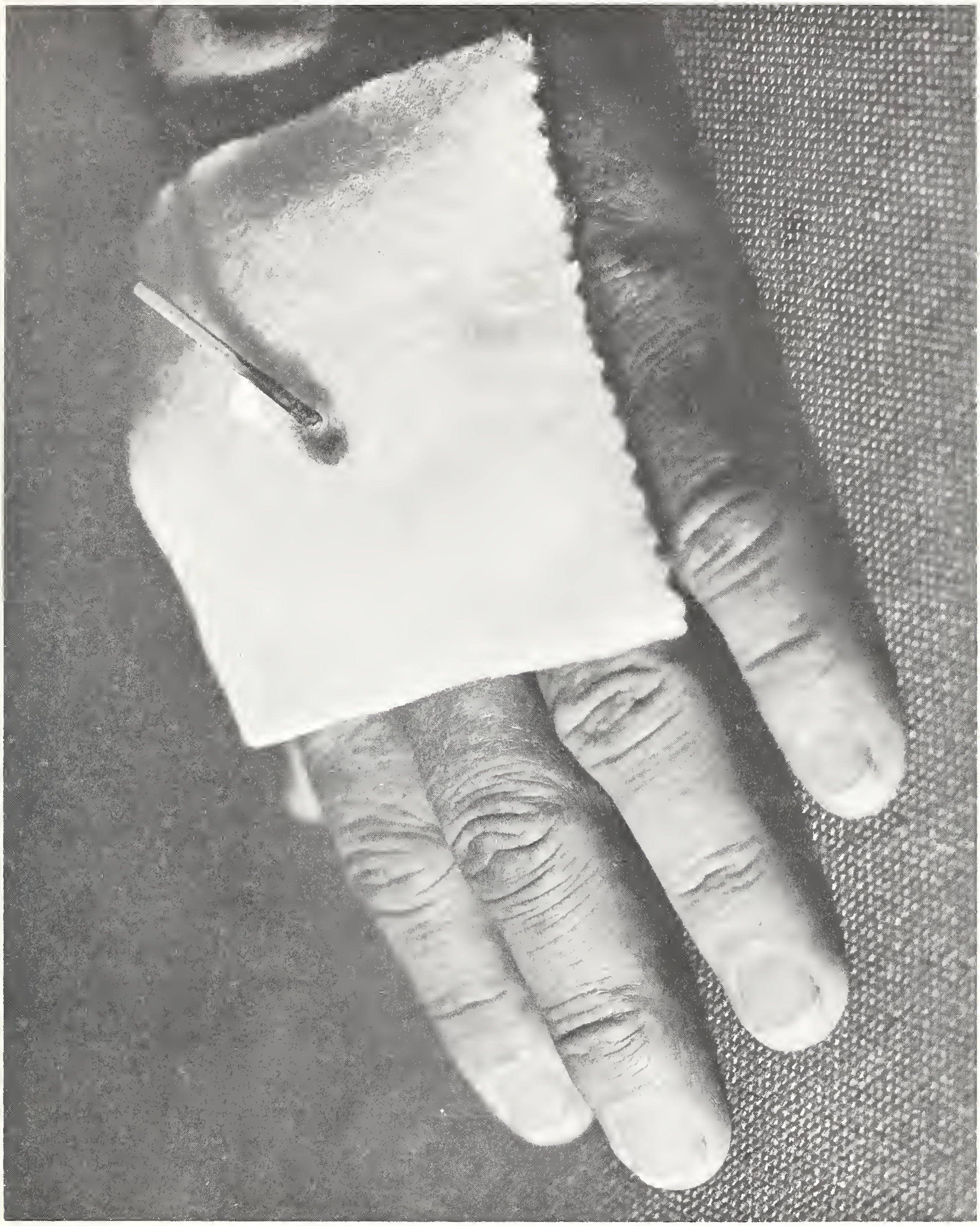

Figure 1.—Illustration of protection provided by all cotton fire resistant fabrics.

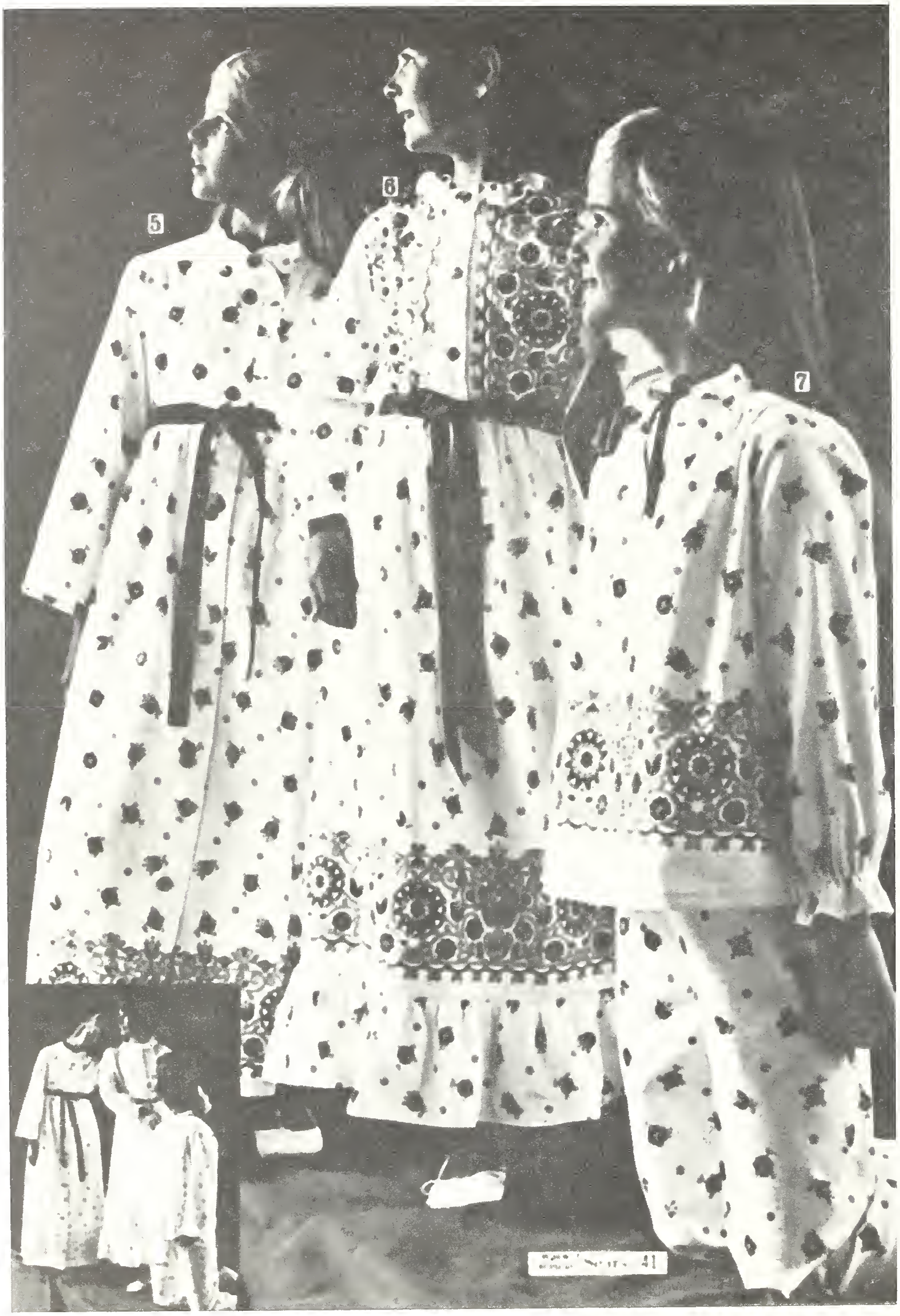

Figure 2.—Illustration of cotton garments treated for fire resistance.

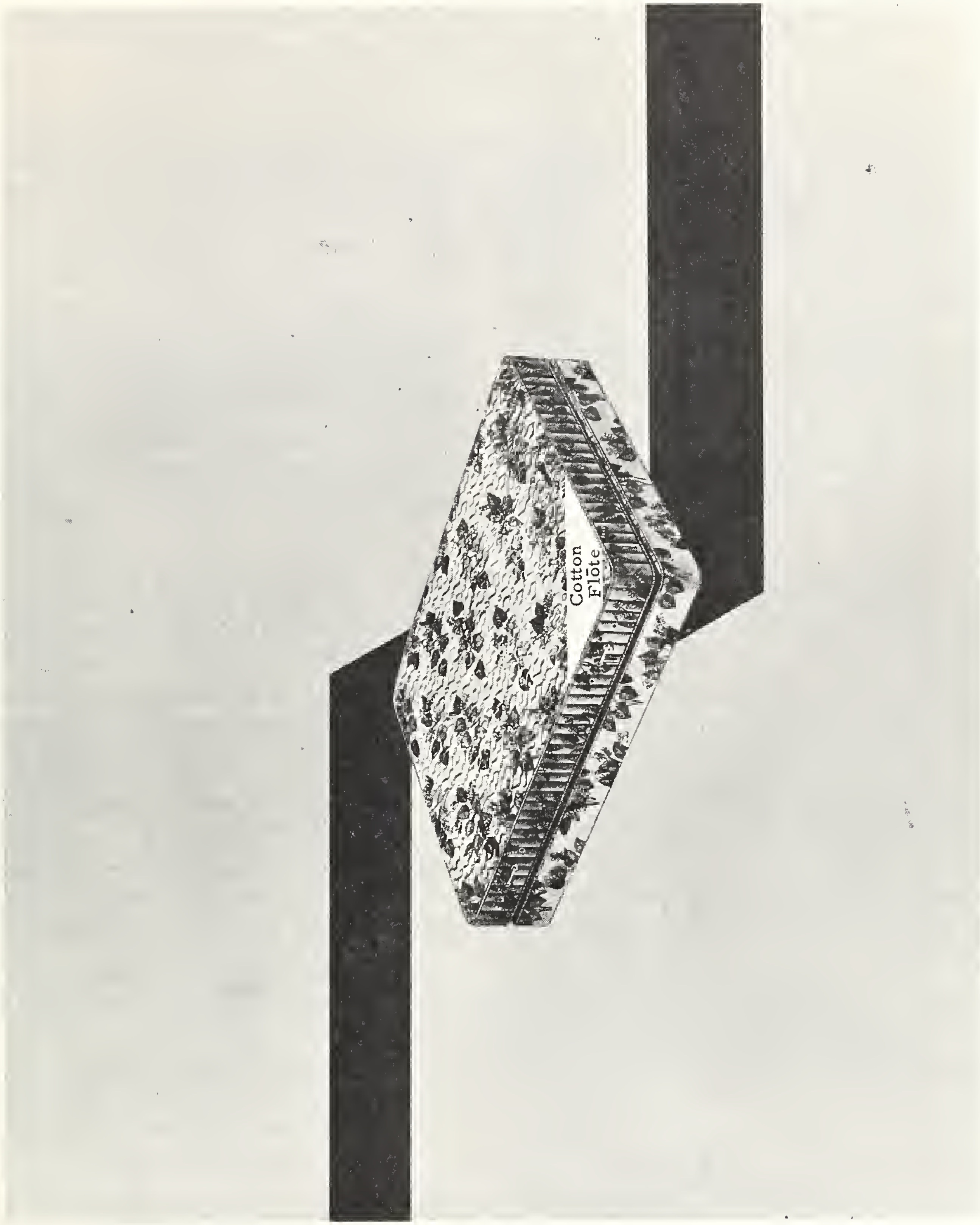

Figure 3.—Flame resistant mattress.

SUMMARY

To assist your further deliberations, I have reviewed and identified the durable fire-resistant chemical processes for cotton fabrics that have competitively survived from the many attempts to develop markets for fire-resistant textiles during the past 35 years. Past industry and government coorporation has also been reviewed as a suggested guide for potential large scale production of improved fire-resistant products for future mass consumer markets.

In conclusion, may I gratefully acknowledge the assistance and cooperation of the following organizations in preparation of the figures and the collection of other information presented to you during this discussion: The Southern Regional Research Laboratory of USDA, The National Cotton Council of America, the Information Council on Fabric Flammability, the American Textile Manufacturers Institute, the U.S. Army Natick Laboratories, and Sears, Roebuck, and Company.

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THEORIES OF FLAME RETARDANCY AND FLAME-RETARDANT UPHOLSTERY FABRICS AND MATTRESS TICKING

by

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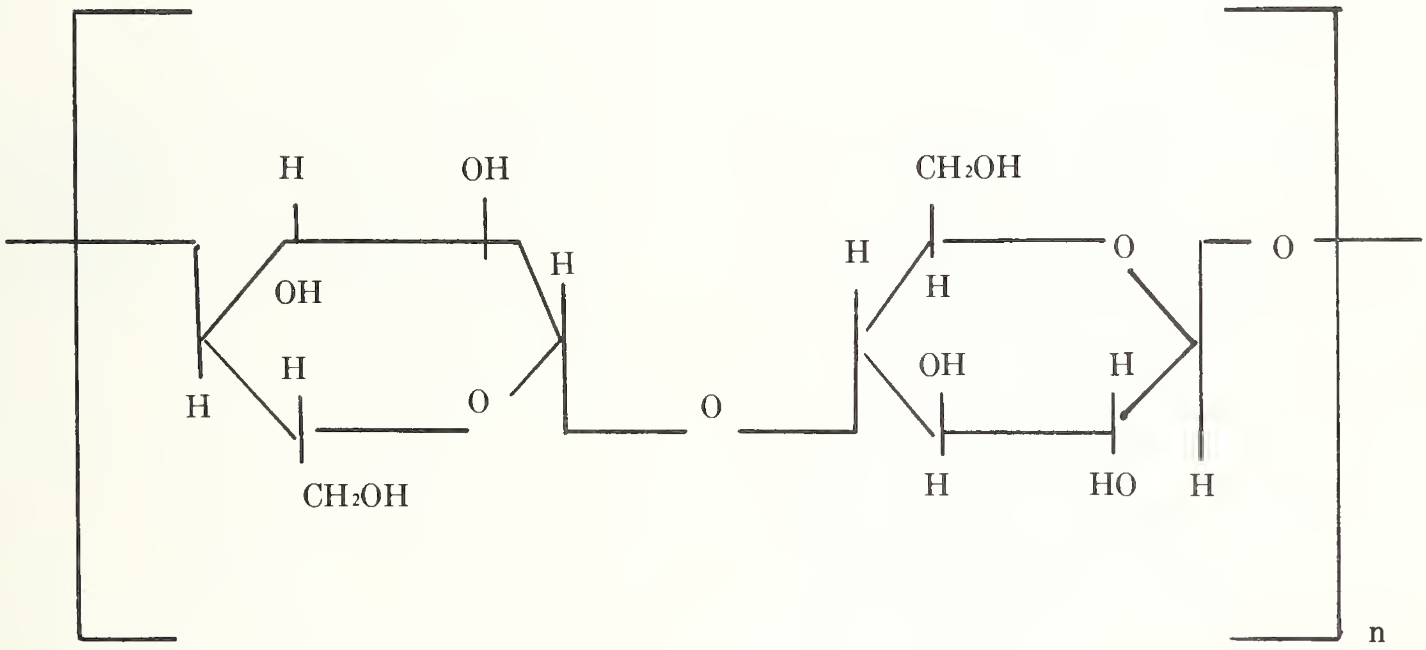
Just think of living in unheated houses! eating raw meat! traveling from place to place without automobiles or airplanes! These thoughts would be realities were it not for the beneficial effects of fire. BUT—fire can also be a terrible enemy if it is not controlled. Every year fire claims many lives; property damage amounts to millions of dollars. Some of these lives could have been saved if fires involving clothing alone could have been eliminated. However, before methods can be devised for the prevention of clothing fires, it is necessary first to understand what happens when textiles burn.

Figure 1.—Cellulose.



The basic building block of cellulose is the beta glucose unit as shown in figure 1. As shown in this figure "n" can vary from 1400 to 2400, depending on the type of cellulose. The burning characteristics of cellulose depend on the quantity and availability of oxygen, as well as other factors. During burning, volatile gases, tars, char, and water are formed. The quantity of tar and char is small and when untreated cellulose burns, the main decomposition products are carbon dioxide (CO_2) and water (H_2O).

Burning, which takes place in two stages, may originate from either an open flame or a hot surface (above 300 ° C); in the first stage, thermal decomposition results in gaseous, liquid, tarry, and solid products. Combustion of the flammable gases takes place. The liquids and tars volatilize, and some of these volatile fractions burn, while portions give a carbonized residue that does not burn readily. This process continues until only carbonaceous matter remains. Then the second stage, or glowing stage, begins. The residual carbonaceous matter oxidizes, glows, and continues to glow until all organic matter is consumed and only a fluffy ash is left. The temperature of burning celluloses and afterglow temperatures are shown in table 1.

Table 1.—Temperature within burning cellulose.

Type of cloth	Flaming	Afterglow
	°C	°C
Cotton-viscose cloth	444	600
Cotton cloth	413	600
Partially acetylated cloth	416	600

The theories that have been proposed to explain the function of a flame retardant fall into the following classes:

(1) coating
(2) gas
(3) thermal
(4) chemical

(1) *Coating theory.*—According to the coating theory, an effective flame-retardant compound forms a glassy skin and stable foam on the fiber surface. Such a flame-retardant coating protects the fabric from air by serving as a barrier to the flame and by entrapping the volatile tars evolved during combustion. Three requisites of the applied compound are: (a) It must decompose at a relatively low temperature; (b) there must be an evolution of large amounts of gaseous products (80 to 200° C); and (c) the gas phase must be nonflammable. The flame-retardant coating must melt at a temperature near that at which gas evolution begins, be minerallike in structure, and be stable at 500° C.

(2) *Gas theory.*—This theory states that the flame retardant decomposes at burning temperatures to give gases which do not burn, but which dilute the flammable gases produced by the decomposition of the cellulose to a concentration below the flaming point. The flame retardant must decompose during burning to produce various nonflammable vapors to such an extent that its presence makes it impossible for gases to flame. Effective gases are carbon dioxide (CO_2), methane (CH_4), hydrogen chloride (HCl), water (H_2O), and sulfur dioxide (SO_2), which result from the thermal decomposition of compounds such as Na_2CO_3, $NaHCO_3$, and NH_4Cl. This theory is obviously not capable of general application since many excellent flame retardants, such as titanium dioxide, do not evolve gases on heating. However, these gases may be of importance in preventing flaming of the flameproofing agent.

(3) *Thermal theory.*—Two mechanisms have been proposed for the retardation of flaming of cellulose. In one, it is proposed that the caloric input from a source is dissipated by an endothermic change of the retardant such as fusion and sublimation of the flameproofer. Such endotherms prevent propagation of the flame. This mechanism is applicable only to those compounds capable of fusing or subliming but not to the majority of known flameproofers. In the second mechanism, it is proposed that the heat supplied from the source is conducted away from the fibers so rapidly that the fabric never reaches combustion temperatures. Because of the small amount of flameproofing agents required in some treatments, this mechanism is obviously not applicable for most flame retardants.

(4) *Chemical theories.*—(a) Hydrogen bonding theory:—It has been suggested that a majority of the known flame retardants are strong hydrogen-bonding agents. Therefore, when water, the bridging medium between cellulose hydroxyls, is lost at high temperatures, the bridges may be maintained by the strong hydrogenbonding activity of the flame retardant, thus stabilizing cellulosic fragments by reducing their volatility and consequently reducing their combustibility. Although this mechanism may be a factor, it must be rejected because the strength of hydrogen bonds depends upon the electronegativity of the atoms involved; even the strongest bonds, such as the fluoride-hydrogen-fluoride bond, have bond energies of only 9 to 10 KCal per mole. Weaker hydrogen bonds (4 to 6 KCal per mole) would be formed with most flame retardants. It is not likely that such bonds could exist at 400° to 500° C.

(b) Catalytic dehydration of cellulose.—Although all theories proposed are of some value, the most important proposes the alteration of the cellulose decomposition path so that the amount of flammable tars and gases is reduced and catalytic dehydration occurs. To propogate a flame or to produce an afterglow, the burning reaction must be exothermic. Comparison of heats of formation of carbon monoxide (CO) and carbon dioxide (CO_2) in figure 2 shows that almost four times as much heat is evolved when a given quantity of carbon is oxidized to

CO_2 than to CO. When the reaction is stopped at CO, the combustion is not self-propagating. Consequently, one method of imparting fire resistance to cotton is to supply a reagent which will react with CO as soon as it is formed, and which will, in effect, be a CO sink. A better approach would be to apply an excellent dehydrating reagent to cotton so that the reaction would proceed to yield only carbon and water. Then the fabric would not burn.

Figure 2

$$\begin{tikzcd}
C+O_2 \arrow[r, "26.4\text{ K.Cal.}"] \arrow[d, "94.3\text{K Cal.}"] & CO + \frac{1}{2}O_2 \arrow[dl, "67.9\text{ KCal.}"] \\
CO_2 &
\end{tikzcd}$$

$$\begin{tikzcd}
 & \text{Volatile gases} \\
\text{(1) Cellulose} + \Delta \arrow[ur] \arrow[r] \arrow[dr] \arrow[ddr] & \text{Tar} \\
 & \text{Char} \\
 & \text{Water}
\end{tikzcd}$$

$$\text{(2) Cellulose} + \Delta \xrightarrow{\text{XS Air}} CO_2 + H_2O$$

$$\text{(3) Cellulose} + \Delta \longrightarrow C + H_2O$$

Nearly all organic materials (including cotton) will burn or char if subjected to a flame. However, cotton is considered flame retardant if it does not propagate a flame when the source of ignition is removed.

Table 2.—Char formation—THPC treated cotton

Percentage add-on	Percentage char
7	25
23	36
28	43
Theoretical percentage char	44.4

In table 2 are data obtained with an efficient fire retardant tetrakis(hydroxymethyl)phosphonium chloride (THPC). It can be seen that an increase in the add-on of THPC causes an increase in the char content of the fabric that ultimately approached the theoretical value.

The Lewis Acid Theory (proposed by Schuyten and others) is the most widely accepted theory. It states that the flame retardant forms effective Lewis acids which hydrolyze the cellulose and dehydrate the glucose units to form carbon and water. The following facts can be explained by this theory: (1) It is difficult to impart flame resistance to cellulose acetate. Acetic acid rather than water would be formed by the Lewis Acid dehydration of cellulose acetate. (2) Because of catalytic dehydration, flame-retardant cottons decompose at a lower temperature than untreated cotton cellulose. The better flame-retardant cottons decompose at about 275 to 325 ° C, whereas the untreated cottons decompose around 450 ° (3) Compounds containing halogens are good flame retardants because they dilute the concentration of flammable gases so that flaming of gaseous byproducts is greatly hindered. They lower the decomposition temperature of the cellulose, and promote hydrolysis. Fluorine is an exception, due to its high heat of formation. (4) Some metal salts act as flame retardants, but unfortunately they catalyze CO_2 formation and this causes afterglow.

(c) The levoglucosan theory:—This theory, proposed by Esteve, suggests that by preventing the formation of levoglucosan which is the major part of the flammable tar produced by heating cellulose, or by altering the path of cellulose decomposition to yield more char and less tar, flame resistance can be obtained. Pascu showed that mesylation or tosylation of cellulose followed by iodination or bromination resulted in a flame retardant cellulosic product. Perhaps the halo cellulose is unable to form levoglucosan. Other cellulose esters such as phosphates or sulfates are also flame retardants.

Examination of the decomposition products of burning textiles revealed that the tar from flame-retardant cellulose was similar to that of untreated cellulose. Thus, reduced flammability is due to a reduction in the combustibles formed rather than the formation of different chemical decomposition products. A correlation exists between the flame resistance of a fabric and the ratio of char:tar or the ratio of $CO:CO_2$ on degradation. The higher the $CO:CO_2$ ratio or the char:tar ratio, the better the flame resistance. Many compounds, including HCHO, acetaldehyde, formic acid, acetic acid, lactic acid, glycollic acid, glyoxal, methanol, and free radicals, have been detected in the slow pyrolytic degradation products of untreated cellulose. Over 30 different products have been isolated and identified. It must be emphasized that much of the work has been under conditions of slow pyrolysis and the decomposition products depend on the rate of decomposition.

Summary.—The best working hypothesis used to explain the flame retardance of cellulose can be stated as follows:

(1) Flame retardancy results from catalytic dehydration of the cellulose through the reaction of the flame retardant with the cellulose.

(2) The flame retardant must be present or formed in situ from its precursor at a temperature close to that of burning cellulose and yield carbon and water rather than highly flammable residues.

(3) The flame retardant must be volatile at 300° to 500° C.

(4) The flame retardant must be a Lewis acid or capable of forming a Lewis acid at the temperature of burning cellulose.

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FLAME RETARDANT COTTON FLOTE

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INTRODUCTION

Commencing in July 1961, research to improve the resilience, dimensional stability, and tensile strength of cotton batting was undertaken at the Southern Regional Research Laboratory under the joint sponsorship of Textile Fibers and By-Products Association, National Cottonseed Products Association, Inc., National Cotton Batting Institute, Foundation for Cotton Research and Education, and Southern Utilization Research and Development Division (SURDD). We are deeply grateful to our sponsors for their support of this research and for their helpful suggestions and guidance.

The product that evolved from this research has been trade named "Cotton Flote" by the National Cotton Batting Institute. I will now give a brief description of the process by which it is made and the physical properties it imparts (fig. 1).

Little change has been made in the conventional batting equipment. First-cut linters and textile waste are open and blended, the trash is removed and then the blend is garnetted. It is at this point that the additional equipment is added. We have found that the chemical can be applied most advantageously by spraying the cotton as it is being discharged from the garnett in the form of a thin web of parallel fibers.

The chemical formulation generally consists of a 50/50 mixture of thermosetting and thermoplastic resins. The thermosetting resin reacts with the hydroxyls of the cellulose to form intrafiber crosslinks that enhance the fiber's inherent resilience. Examples of acceptable thermosetting resins include methylol melamines, modified urea-formaldehydes, and dimethylol dihydroxyethyleneurea. The thermoplastic resin's main function is to achieve adequate interfiber binding to give the batting acceptable tensile strength and dimensional stability. Polymers of vinyl acetates, vinyl acrylates, vinyl chlorides, and styrene-butadiene all perform satisfactorily. Because the spray formulation is an aqueous medium which contains reactive species, a drying and curing oven is necessary. Through-circulation drying can be accomplished by exposure to 200° F.-air for 2 to 3 minutes, and proper curing is accomplished in 1 1/2 minutes with 300° air.

Table 1 shows typical performance data of various densities of Cotton Flote. The interesting thing to note here is the improved resiliency even at the lowest density. Untreated cotton batting with a density of 2 pounds per cubic foot would be expected to show only 70 percent recovery at 60 percent R.H. and 50 percent recovery at 100 percent R.H. The improvement is especially dramatic at the high humidity.

Table 1.—Typical performance data on Cotton Flote products

Density, lb./cu/ft. Item	Min.	1.5	1.8	2.0	2.2	2.5	3.0	3.8
(50 cycles at 1.0 lb./sq. in.) 60 percent R.H.	4	81.2	89.1	93.2	96.1	96.3	97.0	98.0
Percent recovery from deformation loading	0	53.7	70.2	75.0	77.0	79.2	85.2	90.3
(50 cycles at 1.0 lb./sq.in.) 100 percent R.H.	4	60.0	75.6	79.2	81.2	84.0	90.3	92.0
Heat set under load	30	66.6	60.0	57.9	51.7	48.3	42.2	39.0
	120	59.2	55.5	52.0	42.7	39.0	34.6	28.8
Percent compression under 1.0 lb./sq.in. load		70.6	66.7	64.8	59.2	52.9	46.7	43.8

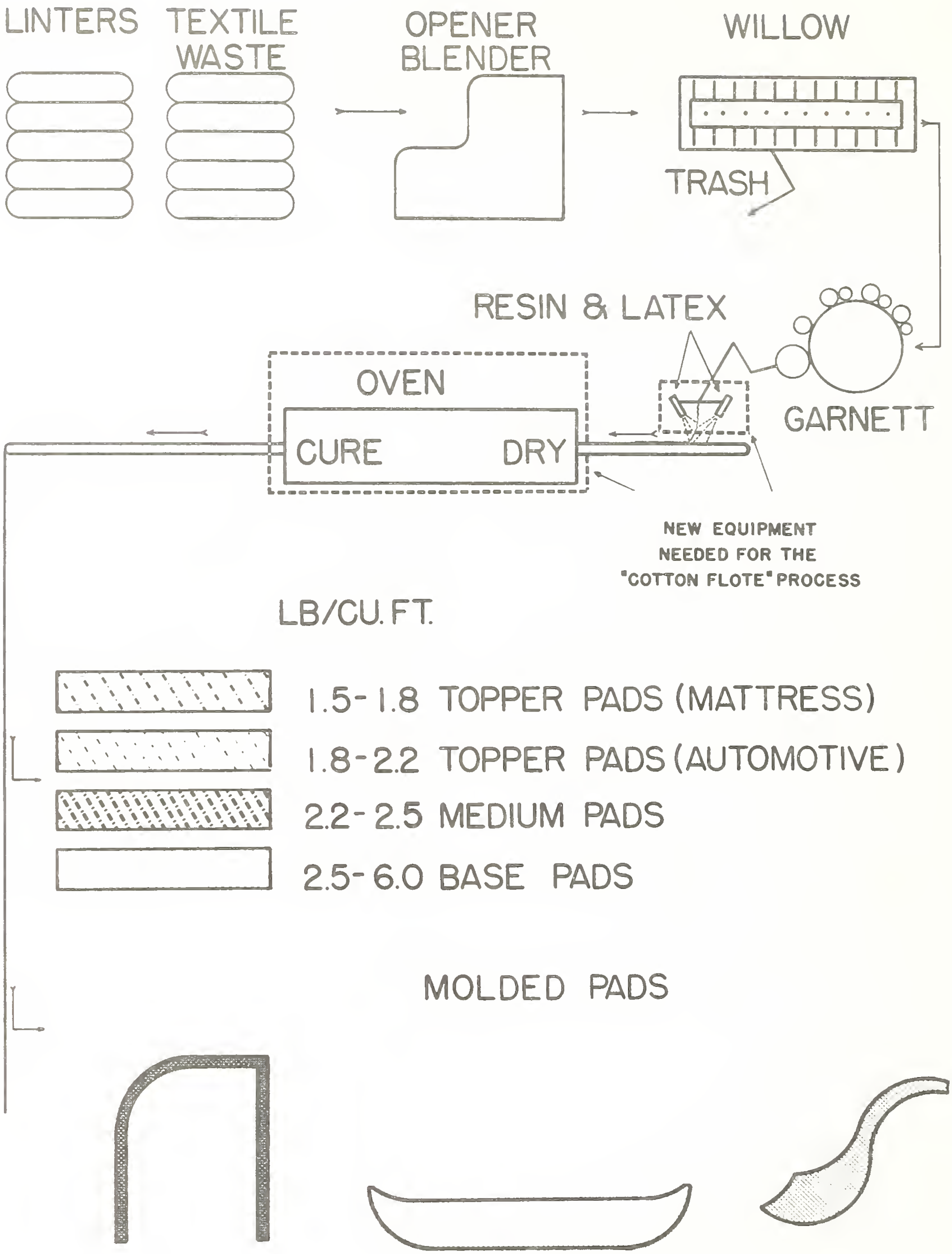

Figure 1.—"Cotton Flote" process.

Table 2 shows typical tensile strength data for various densities of Cotton Flote. Such high strengths as these are unheard of with conventional cotton batting.

Table 2.—Tensile strength of Cotton Flote 1 inch thick

Density	Machine direction	Traverse direction
Lb./cu.ft.	*Lb. to break*	*Lb. to break*
1.0 - 1.5	7.5	4.5
1.5 - 1.8	10.0	6.0
1.8 - 2.2	15.0	10.0
2.2 - 3.0	25.0	15.0
3.0 - 4.5	40.0	25.0
4.5 - 6.0	65.0	35.0

A third dimension—safety.—The process as described to this point has improved cotton batting in two important aspects: (1) strength and (2) stability. The improved strength allows easier handling with lower installation costs and guarantees longer service life. The improved stability guarantees resilient recovery even at high humidity and prevents shifting and clumping. But while strength and stability are two important features of Cotton Flote, there is yet another dimension, and that third dimension is safety. Whether used in automobiles, furniture, mattresses, insulation, or markets yet undiscovered, flame-retardant Cotton Flote should safeguard the consumer.

Let us see how we can achieve this third dimension —safety.

First, let's look at a few essential criteria, other than the obvious requirement that the treatment retard the propagation of flames, which we can use as a guideline for selection of suitable flame retardant treatments. The criteria are:

1. *Inexpensive*—A very important consideration at any time.
2. *Applicable from a water system*—This is mandatory under existing conditions.
3. *Effective on linters and textile wastes*—As long as these are the raw materials, we will have to consider the waxes, pectins, and trash that may interfere.
4. *Compatible with currently used chemical systems* —The key word here is currently.
5. *Durable to mild leaching, high humidity, and temperature*—The degree of durability and the conditions will be determined by end use requirements.
6. *Effective at low levels of chemical add-on*—This is intimately tied to point number 1.
7. *Odor free after drying and curing*—This will be true for any end use.
8. *Effective in reducing afterglow*—This is every bit as important as retarding flames.
9. *Physiologically inactive*—This would be true for just about any end use.

In addition, the treatment must not:

1. *Be deliquescent*
2. *Adversely affect the resilience or tensile strength of products*
3. *Adversely affect the moisture absorption—desorption of fibers*
4. *Adversely affect the air permeability of the array.*

These then are the essential criteria we have used in evaluating flame-retardant treatments for Cotton Flote.

Test methods.—Before examining the results of experimental products that meet these criteria, it may be beneficial to say a few words concerning the test methods we used to evaluate our samples. Unfortunately, there is no generally accepted or standard test for measuring flame resistance of cotton batting. One of the guidelines we used was the General Motor Test Method No. 30-27. This method is a vertical test using a wooden match as the source of ignition.

Another test method we used was the AATCC Standard Test Method 34-1966 (Fire Resistance of Textile Materials). This is also a vertical test. Fortunately at SURDD, our Textile Testing Group has the physical apparatus necessary for this test. The important feature of this apparatus is a timer which automatically extinguishes the bunsen burner flame after 12 seconds. The use of this timer and bunsen burner allows us better reproducibility than we could achieve using a wooden match. Figure 2 shows a photographic mock-up of the apparatus. By using a modification of AATCC 34-1966, we are able to measure afterflame, afterglow, and char length.

We have also examined the use of the Methenamine Timed Burning Tablet as a means of simulating a burning cigarette as a source of ignition. In this test, a tablet is placed in the center of the sample and ignited by touching a match carefully to the edge of the tablet to avoid contacting the surface of the batting. We found that this test is not very

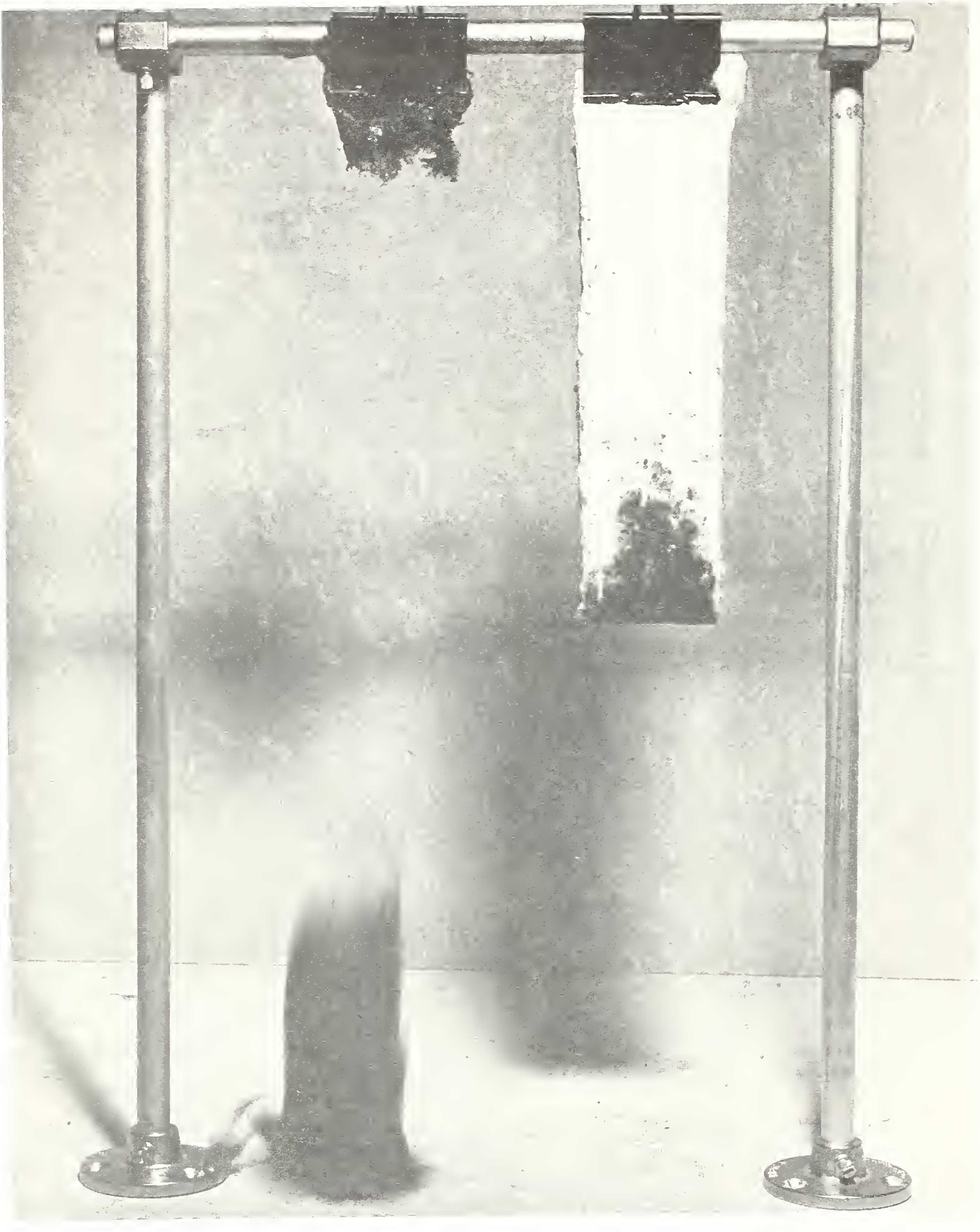

Figure 2.—Mock-up of apparatus for measuring flame resistance of cotton batting.

sensitive. Not only was it impossible to measure the effect of flame retardant concentration, it was even difficult to notice much difference in performance between flame-retardant and nonflame-retardant Cotton Flote. We feel that this may be due to the fact that the temperature at the cotton-flame interface is only 450 ° to 500 ° F and the char formed provided sufficient insulative properties to prevent further degradation. The hot portion of the flame, approximately 750° F., was in the atmosphere above the sample. In the vertical test, the hottest portion of the flame impinges on the cotton, and this temperature is apparently greater than the minimum needed for adequate treatment defferentiation.

I would also like to point out that the vertical test that we use is for screening purposes only, and we do not want to give the impression that it measures all the fire-retardant properties that may be important.

Chemical evaluation.—A. *Borates.*—Boron-containing compounds are a good place to start our discussion of flame retardants for Cotton Flote. The borates have long been known for their ability to impart flame retardancy to cotton and we expected that they would do fairly well in a Cotton Flote formulation.

Table 3.—Chemical composition and resilience characteristics

Item	Sample no. S	T	U
Thermosetting resin	DMDHEU[1]	MMM[1]	MMM[1]
Percent add-on	10	6	7
Thermoplastic resin	ACRY[1]	ACRY[1]	PVC[1]
Percent add-on	10	6	10
Flame retardant	NABOR[1]	NABOR[1]	NABOR[1]
Percent add-on	5	8	3
Density, lb./cu.ft.	2.7	2.3	2.3
Cyclic loading recovery[1]			
65 pct. R.H.	96.5	94.7	95.2
100 pct. R.H.	75.8	84.2	71.4
Percent compression	64.5	63.1	58.3

[1] See page 105 for explanation of symbols.

[2] After 4 minutes.

Table 3 shows the chemical composition and resilience characteristics of formulations containing sodium borate. The important point to note here is that even at the highest level of borate (8 percent), we haven't impaired product performance. Notice that there are four different means of conditioning the samples before testing. The inability of sodium borate to completely inhibit afterglow is quite evident. It also appears that the presence of moisture accentuates this problem. Sample U, you will notice, performs best and this may at first seem surprising if you consider that it has the lowest add-on of sodium borate (3 percent). However, this sample also has a 10-percent add-on of PVC as the latex portion of the formulation. I might also mention here that we looked at zinc borates which are much less water soluble than the sodium salts and hence should perform better at high humidity. However, the zinc salts were so insoluble we couldn't spray the formulation satisfactorily.

B. *Polyvinyl chloride latex.*—Perhaps now would be a good time to discuss our findings concerning polyvinyl chloride. As you all know, polyvinyl chloride homopolymer is considered to be inherently flame-retardant. However, copolymers and plasticized homopolymers do not necessarily perform as well as the homopolymer. We formed batts containing, 5-, 10-, 15-, and 20-percent polyvinyl chloride homopolymer and found that while it inhibited afterglow considerably and to a lesser extent reduced afterflame, polyvinyl chloride without other flame retardant additives did not produce a product we would classify as flame retardant. As you have already seen, PVC works quite well in combination with sodium borate. We will see later that it is effective in other systems as well.

C. *Acrylate latexes with flame retardant salts.*—In general, we have found that products containing vinyl acrylate latexes give better resilience performance than do products containing PVC. It is for this reason that there may be some advantage in using a fire retardant system with a latex other than PVC.

D. *Antimony trioxide.*—Antimony trioxide has also been examined by the research team. In order to use this water insoluble flame retardant it must be compounded or pigmented and then dispersed in the formulation. This procedure can be a bit tricky for the inexperienced, but fortunately antimony trioxide is commercially available already dispersed in polyvinylidene chloride. The sample we examined contained equal parts of polyvinylidene chloride and antimony trioxide. A 10-percent add-on of this, along with an equal add-on of melamine, produced a sample with adequate flame resistance. This flame retardant is a bit more expensive than others we have looked at, but this product because of its insolubility performed better at high humidity and saturation.

Flame retardant formulations containing acrylate latexes are shown in table 5. EB 3067, used in sample 604 is a thermosetting acrylic especially compounded to have a high tolerance for borax and other salts. EB 3074, found in samples 602 and 603, is identical to EB 3067; however, 40 parts of diammonium phosphate (DAP) and 10 parts of borax have

Table 4.—Flame retardancy characteristics

Item		S	T	U
70 ° F.—65 pct. R.H.:				
Afterglow	sec.	6	7	1
Overall char	cm.	2	2	3
100°—pct. R.H.:				
Afterglow	sec.	15	3	2
Overall char	cm.	3	2	3
158°—16 hr. (dry):				
Afterglow	sec.	5	6	0
Overall char	cm.	3	4	8
158°—16 hr (wet):				
Afterglow	sec.	12	4	0
Overall char	cm.	4	2	4

Table 5.—Chemical composition and resilience characteristics

Item	Sample no.			
	602	603	604	628
Percent solids in spray	20	20	20	25
Thermosetting resin	CB			A-803[1]
Percent of solids	50			35
Thermoplastic resin	EB 3074[1]	EB 3074[1]	EB 3067[1]	CL-304[1]
Percent of solids	50	100	100	35
Density, lb./cu.ft.	2.0	2.3	2.3	1.9
Cyclic loading recovery[2]:				
65 pct. R.H.	90.3	84.8	86.6	92.5
100 pct. R.H.	73.0	69.5	71.0	64.8
Percent compression	62.6	64.7	59.2	63.0

See page 105 for explanation of symbols.

[2] After 4 minutes.

Table 6.—Flame-retardancy characteristics

Item		Sample No.			
		602	603	604	628
100°—65 pct. R.H.:					
Afterglow	sec.				
Overall char	cm.				
100°—100 pct. R.H. (3 days):					
Afterglow	sec.	0	0	B.E.L.[1]	0
Overall cha	cm.	5	10	B.E.L.[1]	4
158°—16 hr. (wet):					
Afterglow	sec.	6	6	B.E.L.[1]	1
Overall char	cm.	9	13	B.E.L.[1]	6

[1] B.E.L.—burned entire length.

been added. (This is based on the latex solids.) Sample 628 contains 35 parts acrylate, 35 parts modified urea formaldehyde and 30 parts of a mixture of DAP-borax-urea in a weight ratio of 3:1:1. Notice that to get adequate resilience performance a crosslinking resin is necessary. Table 6 shows the flame retardancy characteristics. In comparing samples 602 and 603, it would have to be said that they are about equally effective. However, sample 602 contains exactly half the flame retardant. Here we feel the presence of the nitrogen containing resin, namely the melamine in sample 602, acts synergistically with the flame retardant. We have noticed this in numerous samples and would say that it is not just a chance occurrence. Other researchers at SU have noticed a similar effect when treating fabric with flame retardants. Sample 604 demonstrates that the acrylate without flame retardant has no flame retarding properties. Sample 628, containing a laboratory prepared flame retardant, shows superior performance under the water saturation test. Literature has indicated that the presence of urea in a DAP formulation inhibits the ion exchange properties that diammonium phosphate usually exhibits. In hard water the ammonium ions are exchanged for calcium ions and the calcium phosphate formed is not an effective flame retardant. This may be occurring in the case of samples 602 and 603 where no urea is present.

E. *Phosphorus-containing flame retardants.*—Finally, I would like to describe the results of our evaluation of phosphorus-containing flame retardants. Phosphorus has been applied to cotton in various chemical forms, for example, phosphoric acid, diammonium phosphate, urea phosphate complexes, and various reaction products of phosphoric acid and organic compounds. While a great deal of theory has been developed concerning phosphorus flame retardants, I won't go into the theory at this time. Instead, I will show results of commercial flame retardants that were developed with such theoretical considerations in mind. Table 7 presents formulations containing a urea phosphate complex as the flame retardant. Notice the excellent recovery characteristics, especially at 65 percent R.H. Sample 013 was sprayed with only enough acrylate latex to give minimum binding and shows that this urea phosphate complex improves the resilience of the product over that of untreated cotton.

The flame retardancy characteristics are shown in table 8. The first two samples show a tendency toward afterglow. Both of these samples contained 2-percent urea phosphate. Sample 652 contained 4-percent urea phosphate and definitely inhibited the afterglow and shortened the char length. The effect of the combination of urea phosphate and PVC is

Table 7.—Chemical composition and resilience characteristics

Item	Sample no.			
	650	651	652	103
Thermosetting resin	MMM[1]	DMDHEU[1]	MMM[1]	
Percent add-on	9	9	8	
Thermoplastic resin	ACRY[1]	ACRY[1]	ACRY[1]	ACRY[1]
Percent add-on	9	9	8	1
Flame retardant	UP[1]	UP[1]	UP[1]	UP[1]
Percent add-on	2	2	4	9
Density, lb./cu.ft.	2.2	2.0	2.8	2.1
Cyclic loading recovery[2]:	98.2	94.8	96.3	85.7
65 pct. R.H.	78.2	68.9	74.1	75.5
100 pct. R.H.				
Percent compression	61.3	63.6	50.0	73.2

F—Flashed to top.

[1] **See page 96 for explanation of symbols.**

[2] **After 4 minutes.**

Table 8.—Flame retardancy characteristics

Item		Sample no.			
		650	651	652	013
70° F—65 pct. R.H.:					
Afterglow	sec.	5	5	0	2
Overall char	cm.	10	4	4	2
100°—100 pct. R.H. (3 days):					
Afterglow	sec.	2	1	0	0
Overall char	cm.	7	6	4	5
158°—16 hr. (dry):					
Afterglow	sec.	1	1	0	2
Overall char	cm.	8	5	4	4
158°—16 hr. (wet):					
Afterglow	sec.	1	3	1	0
Overall char	cm.	9	6	5	4

shown in table 9. The resilience performance is good but not quite as good as with acrylate. Improvement in the inhibition of afterglow, however, is indicated in table 10. Remember that the urea phosphate concentration lies between 2 and 4 percent for these samples.

The data presented in the past four tables suggests that a good compromise might be the use of a vinyl chloride/acrylate copolymer. This may give the best balanced performance.

Table 11 shows our evaluation of a product formed by the reaction of dicyandiamide and phosphoric acid. It was dimethylolated to improve its stability and enhance its reactivity with cellulose. Here again the resilience properties are excellent.

Table 9.—Chemical composition and resilience characteristics

Item	Sample no.			
	589	587	586	609
Thermosetting resin	MMM[1]	MMM[1]	MMM[1]	MMM[1]
Percent add-on	10	9.6	10	8
Thermoplastic resin	PVC[1]	PVC[1]	PVC[1]	PVC[1]
Percent add-on	8	7.6	6.8	8
Flame retardant	UP[1]	UP[1]	UP[1]	UP[1]
Percent add-on	2	2.8	3.2	4
Density lb./cu.ft.	2.8	2.2	2.1	2.1
Cyclic loading recovery[2]:				
65 pct. R.H.	87.2	94.0	92.8	91.5
100 pct. R.H.	78.7	68.0	70.6	68.0
Percent compression	52.9	60.7	61.0	65.4

[1] See page 105 for explanation of symbols.
[2] After 4 minutes

Table 10.—Flame retardancy characteristics

Item		Sample no.			
		589	587	586	609
70° F—65 pct. R. H.:					
Afterglow	sec.	0	0	0	0
Overall char	cm.	7	10	9	6
100° —100 pct. R.H. (3 days):					
Afterglow	sec.	0	0	0	0
Overall char	cm.	8	5	4	5
158° —16 hr. (wet):					
Afterglow	sec.	0	0	0	0
Overall char	cm.	7	10	11	5

Table 11.—Chemical composition and resilience characteristics

Item	Sample no.		
	131	132	133
Thermosetting resin	MMM[1]	MMM[1]	MMM[1]
Percent add-on	8.5	7.5	6.5
Thermoplastic	ACRY[1]	ACRY[1]	ACRY[1]
Percent add-on	8.5	7.5	6.5
Flame retardant	O-P	O-P	O-P
Percent add-on	3	5	7
Density lb./cu.ft.	3.4	2.7	3.0
Cylic loading recovery[2]:			
65 pct. R.H.	93.8	94.3	96.2
100 pct. R.H.	87.5	75.5	75.0
Percent compression	50.0	57.1	48.1

[1] See page 105 for explanation of symbols.
[2] After 4 minutes

Notice in table 12 that 5 percent seems to be the minimum effective flame retardant concentration. Sample 131, with only 3 percent flame retardant, exhibits considerable afterglow. Samples 132 and 133 indicate that this flame retardant remains quite effective even after exposure to high humidity or saturation with water if the flame retardant concentration is above 5 percent.

Different densities.—As you may have noticed, all the data presented up to this point relate to low density Cotton Flote products. We have, however, evaluated some flame retardant formulations at higher densities.

Table 13 shows the formulations used. Sample J was our attempt at a product of over 6 lb./ft.^{3}. Notice that the total solids for this formulation is 40 percent and that the ratio of thermosetting to thermoplastic resin is 2:3. The flame retardant is a commercial product described as a borated urea formaldehyde. The add-on was about 4 percent. Sample K, at 4.1 lb./ft.^{3}, used a lab prepared urea diammonium phosphate flame retardant at 6.5-percent add-on. Sample L, a 3.1 lb./ft.^{3} product, used a borated amido polyphosphate at 3-percent add-on as the flame retardant. Sample M, at 2.4 lb./ft.^{3}, used a combination of urea phosphate, polyvinyl, and polyvinylidene chloride for flame retardancy. Sample N and the control were both in the 2-pound per cubic foot range.

Tables 14 and 15 show the flame resistance performance. The poorest performance, although still acceptable by some standards, was found in sample K, the standard lab formulation of urea and diammonium phosphate. By increasing the concentration up to 8 percent, we have improved this performance. Generally speaking, we found that as the product density increased, the add-on requirement decreased for all flame retardant systems.

Table 12.—Flame retardancy characteristics

Item		Sample no.		
		131	132	133
70° F—65 pct. R.H.				
Afterglow	sec.	0	0	0
Overall char	cm.	9	8	10
100° F—100 pct. R.H.:				
Afterglow	sec.	12	0	0
Overall char	cm.	9	6	7
158° F—16 hr. (dry):				
Afterglow	sec.	15	0	0
Overall char	cm.	10	15	11
158° F—16. hr (wet):				
Afterglow	sec.	0	0	0
Overall char	cm.	10	8	5

Table 13.—Spray formulations

Item	Sample no.					Control
	J	K	L	M	N	1
Chemical composition of spray						
Percent solids in spray	40.0	30.0	20.0	20.0	10.0	20.0
Thermosetting resin	DMDHEU	UF	MMM	MMM	..	MMM
Percent of solids	35.8	26.1	40.0	39.8	..	50.0
Thermoplastic resin	PVC	ACRY	ACRY	PVDC	ACRY	ACRY
Percent of solids	53.7	52.2	45.0	32.7	12.0	50.0
Flame retardant	BUF	UDAP	BAP	PVC 8.2 UP	UP	..
Percent of solids	10.5	21.7	15.0	19.3	88.0	..
Catalyst	$ZnCl_2$	NH_4Cl	$MgCl_2$	$MgCl_2$	..	$MgCl_2$
Buffer	..	..	DCY	DCY	..	DCY

Table 14.— Flame resistance performance

Item		Sample no. J	K	L	M	N	Control 1
Conditioned 70° F—65 pct. R.H.:							
Afterflame	sec.	0.0	0.0	0.0	0.0	0.0	3.3
Afterglow	sec.	.0	4.5	.0	.0	2.0	20.8
Overall char	cm.	3.9	4.8	2.5	7.1	2.1	11.7
Main char	cm.	1.5	1.8	1.0	2.9	1.5	7.5
Soaked with H_2O—Dried 158° F.:							
Afterflame	sec.	0.0	6.0	0.0	0.0	0.0	4.7
Afterglow	sec.	.0	5.0	.0	.0	.0	2.5
Overall char	cm.	5.8	9.0	2.5	4.0	4.0	11.1
Main char	cm.	3.3	3.0	1.0	2.0	1.5	8.5

Table 15.— Flame resistance performance

Item		Sample no. J	K	L	M	N	Control 1
100 pct. R. H.—3 days—100° F.:							
Afterflame	sec.	0.0	8.5	0.0	0.0	0.0	8.5
Afterglow	sec.	.0	5.0	.0	.0	.0	46.0
Overall char	cm.	3.3	8.8	2.0	5.0	4.8	7.8
Main char	cm.	1.5	2.5	1.0	1.5	1.6	3.2
158° F—16 hours:							
Afterflame	sec.	0.0	5.0	0.0	0.0	0.0	2.0
Afterglow	sec.	.0	5.0	.0	.0	2.5	5.0
Overall char	cm.	3.9	6.3	2.0	5.0	3.5	16.1
Main char	cm.	1.8	2.3	1.0	1.5	1.0	6.0

CONCLUSIONS

In conclusion, I would like to make some general observations regarding our experiences with flame-retardant systems for Cotton Flote. We have found that the spray distribution and quality of web are important parameters in producing flame-retardant products. A uniform spray pattern insures good chemical distribution and a finer web allows more intimate chemical penetration.

Not only has it been shown that cotton batting can be made flame retardant on a pilot scale, but in addition, at least one company is already marketing a flame-retardant Cotton Flote innerspring mattress and box spring combination. The flame-retardant Cotton Flote is a direct result of this research, while the ticking treatment is based on another phase of cotton flame retardancy research conducted at the Southern Regional Research Laboratory.

Finally, it has often been said that "the difference between stumbling blocks and stepping stones is the use we make of them." Let us hope that we can use flame-retardant Cotton Flote as a stepping stone to increased safety for the consumers and increased utilization of cotton for the producers.

Identification of symbols used in tables

Symbol	Chemical nature
ACRY	Acrylate latex
A-803	Modified urea formaldehyde
BAP	Borated amido polyphosphate
CB	Melamine
CL-304	Reactive acrylate
DMDHEU	Dimethylol dihydroxyethyleneurea
EB 3067	Acrylate latex
EB 3074	Acrylate with 40 parts of diammonium phosphate and 10 parts of borax
NABOR	Sodium borate
MMM	Methylated methylol melamine
O-P	Organo-phosphorus compound
PVC	Polyvinyl chloride
PVDC	Polyvinylidene chloride
UF	Urea fomaldehyde
UP	Urea phosphate
UDAP	Urea diammonium phosphate

SECOND SESSION: S. J. Henry, Jr., Manufacturers Products Division, Southern Cross Industries, Inc., Atlanta, Ga., Chairman

LATEST RESEARCH AT SURDD TO DEVELOP FLAME RETARDANT CONVENTIONAL COTTON BATTING

by

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Most of the research to develop flame retardant Cotton Flote products was carried out during the period from June 30, 1967, to January 30, 1968, after the termination of the cooperative research project, which led to the development of Cotton Flote. A new cooperative research project having the same sponsors was initiated in April 1968. The new work is also receiving support from the National Association of Bedding Manufacturers. The intent of the research is the development of flame retardancy treatments that would be suitable for use in the production of conventional cotton batting.

This morning you have heard Messrs. Sutcliffe and Ryan imply that the 1967 amendments to the flammable textiles act will some day in some way affect mattresses and furniture. The research I am going to discuss anticipates the need for flame resistant conventional cotton batting products.

The requirements for the application of flame retardant characteristics to conventional cotton batting are considerably different than those that apply to the Cotton Flote system, although many of the same chemical compounds can be expected to do the job effectively. For example: In the Cotton Flote process, the bonding of fibers together at points of contact is a desirable characteristic that assists in achieving the tensile strength, dimensional stability, and integrity of the products. In addition, the presence of the thermoplastic resin which serves as the bonding agent makes the removal of the flame retardant chemicals more difficult at high relative humidity and even under conditions of mild leaching. Furthermore, all of the needed chemicals are applied in an operation already incorporated into the process, in a situation where the fibers are in an open array and readily accessible to the treating formulation.

For conventional cotton batting there seems to be no really good place to apply the flame retarding chemicals to the fibers during normal mechanical processing. It appears, therefore, that the flame retardancy treatment must of necessity be applied as a pretreatment before any mechanical processing of the fibers is undertaken. If the fibers are to be garnetted after treatment, the flame retardancy treatment used should not bind the fibers together at points of contact, since such binding would result in the breakage of large numbers of fibers and generally result in an inferior product. Emulsion polymers, such as vinyl chloride, vinylidene chloride, and some acrylic polymers, that contribute some additional flame retardancy and reduce the tendency of the flame retardant to be lost by leaching or high humidity in the Cotton Flote process should probably be eliminated from consideration for the pretreatment of fibers for conventional cotton batting.

Some of the criteria that the flame retardant treatment must meet are shown in Table 1.

Table 1.—Essential criteria for flame retardance treatments for cotton batting rawstock

The treatment must be:

1. Inexpensive
2. Easy to apply
3. Effective on native linters and textile wastes
4. Effective at low levels of chemical add-on
5. Durable to mild leaching, high humidity, and temperature
6. Odor free after drying and curing
7. Effective in reducing after glow
8. Physiologically inactive

The first item is, of course, of prime importance in a finished batting product that is expected to sell for 20 to 40 cents per pound. There are a number of chemical compounds that are known to do an excellent job of rendering cotton flame retardant. Among these are tetrakis(hydroxymethyl)-phosphonium chloride (THPC), tris(l-aziridinyl)phosphine oxide (APO), and tris(2,3 dibromopropyl)phosphate, which were mentioned by George Drake and Larry Heffner this morning. These chemicals sell for approximately $1 per pound and add-ons of 10 percent or more are needed. As a consequence, their use in cotton batting is obviously uneconomical. Alternatively then we need to use resin precursors, resin formers, or compounds that will in some way react chemically with cellulose to give it flame retardancy characteristics. The cost of these materials should not exceed 40¢ per dry pound, and effective add-on levels should be less than 10 percent by weight.

The second item deals with the application of the chemicals. Ease of application can be interpreted in a number of ways—the mechanical system that might be most appropriate, the chemical system and its particular requirements, the handling of the fibers during treatment and subsequent drying and/or curing, followed by the storage of treated fibers. You will be hearing from a number of equipment manufacturers later in this program who will give you their ideas about many of the factors involved. For our experimental purposes, we have limited ourselves to the use of water based chemical systems using immersion followed by squeeze rolls to bring the wet pickup to the predetermined level. Alternative approaches to be considered in the near future include immersion followed by centrifugation, and pressure impregnation during immersion followed by appropriate dewatering techniques.

We have, of course, explored the possibilities of using organic solvents to impregnate the fibers with the chemicals. To be commercially feasible such systems require a solvent recovery unit which would undoubtedly increase the initial capital investment. If the cost is low enough, however, solvent systems do have some possible advantages.

The next item states that the treatments we are seeking should be effective on the cotton fibers as they are received. Stated another way, we want to avoid the necessity of preprocessing the linters and textile wastes to remove waxes, pectins, oils, trash, and other extraneous materials.

Next we need to devise a chemical system that will give us the best performance insofar as flame retardancy is concerned while requiring the smallest amount of chemical add-on possible. Both of these items have a bearing on the cost of the final product and are important in selecting the treatment to be used. The next four items deal with the properties that the treated fibers must exhibit if the products are to be accepted by the consumer. Durability of flame retardancy for cotton batting products is of important, because cotton batting products are used in many and varied environmental conditions. Consequently the products must retain their flame retardancy characteristics under conditions of high temperature, high humidity, and even mild leaching. These requirements practically eliminate the use of such former standby treatments as borax-boric acid, diammonium phosphate, ammonium sulfamate and other inorganic salts.

Obviously when products are destined for furniture or mattresses, or automobiles for that matter, one has to be sure that no undesirable odors are generated by the chemicals used to contribute flame retardancy. Amines, amides, and other chemicals have particularly disagreeable odors and for the great part must be eliminated from consideration. Likewise some phenol derivatives have strong pungent odors; consequently their usefulness is minimized. The last item says that the products we are seeking must not adversely affect the health of the users. Of particular concern would be allergenic reactions and the like for the consumer, and carcinogenic or other potentially serious effects on employees in the manufacturer's plant.

Table 2 outlines some of the things that the treatment must avoid if product performance other than flame retardancy is to be maintained.

Table 2.—Essential criteria for flame retardance treatments for cotton batting rawstock.

The treatments must not:

1. Be deliquescent
2. Reduce quality of webs produced
3. Significantly increase fly during garnetting
4. Affect the moisture absorption-desorption of fibers
5. Affect the air permeability of the array
6. Dust or powder off the fibers

The chemical used should not deliquesce, that is absorb water from the air under various environmental conditions, and cause the product to become soggy and lose resilience. It should not reduce web quality and thus reduce product quality or increase fly during garnetting. As mentioned earlier, this requirement practically eliminates a number of film forming compounds that exert a synergistic or contributing effectiveness to the flame retardancy. It more or less reduces the types of chemicals to be considered to those that are soluble in water, or other solvents, so that the treatment can penetrate into the fibers and leave a minimum on the surface to adhere the fibers together.

While water, at least at present, seems to be the best candidate as a carrier for the treatment, it should be pointed out that the use of an aqueous system introduces other problems. Few chemicals that are initially water soluble become water insoluble or water impervious after drying and/or curing. Consequently, chemical means of enhancing the durability of the treatment to cotton against high relative humidity and even mild leaching must be sought concurrently with flame retardancy characteristics. Resin systems, resin formers, or chemically reactive compounds would seem to be the best answer.

The fourth item is also related to the one just discussed. While we want the treatment to become permanent by attachment to the cotton in some way, we, at the same time, want to be sure that the treatment does not change the native characteristic of the cotton to absorb moisture and to lose moisture with changes in environmental conditions.

Likewise, we do not want the treatment to reduce the resilience, or "deaden the fibers," to cause them to pack down, and thus interfere with the free passage of air into and out of the batting array.

Finally, we want the treatment to be attached to the fibers in such a way that when the fibers are garnetted, the treating chemical does not dust or powder off. Furthermore, we want the treatment to remain with the fibers in the batt during its useful life and not be lost due to migration, sublimation, or any other process.

Now that the problems we face in the development of an effective flame retardant for cotton batting have been outlined, let me tell you something about what we have accomplished during the past 6 months toward solving the problems.

Our first concern was to establish a means of measuring what we considered to be the most important characteristics of a flame retardant and to define what we mean by effective flame retardancy. There are quite a number of test procedures available for the measurement of rate of burning, of flame retardancy and of self-extinguishing characteristics that have been accepted by the textile industry for many years.

Because we believe that for our research products we should select the most demanding test procedures, we chose to evaluate the products using a modification of the American Association of Textile Chemists and Colorists procedure 34-1964. This test involves hanging a sample 2 inches wide by 10 inches long vertically over a bunsen burner. Samples are usually 1 inch thick. The flame from the bunsen burner is 1 1/2 inches high and the sample is adjusted to project 3/4 inch into the flame. The flame burns for 12 seconds. We measure the time that the flame persists after the bunsen burner is extinguished, and the time that the red afterglow persists. We also measure the length of the main charred area at the bottom of the sample and the overall char length including scorch from flashing. With these measurements we can see the differences in effectiveness among a number of treatments to the point where we can, with reasonable confidence, select samples with different degrees of flame retardancy. Since there is also the question of flame retardancy under a number of environmental conditions, we usually test the samples after (1) conditioning at 70° F. and 65 percent R.H. for 24 hours, (2) 100° F and 100 percent R.H. for 3 days, (3) 158° for 16 hours, and (4) soaking with water and drying at 158° for 16 hours. At a later date we also plan to go back and test samples that have been stored in our pilot plant from 6 months to 1 year after manufacture to ascertain if the flame retardancy is being lost.

EXPERIMENTAL

With this background, let me give you a brief résumé of what we have produced in the way of experimental samples. During this presentation I am going to refer to a number of chemical compounds that we have evaluated in our study so far. For simplicity and for your information we are going to use trade names for these chemicals. The use of these trade names should not be interpreted as an endorsement of the products by the USDA over similar products of other manufacturers. Appendix "A" at the end of this paper identifies the manufacturers of the products mentioned.

The usual procedure followed to prepare flame retardant samples was as follows:

A blend of fibers consisting of 60 percent Delta first cut linters and 40 percent of various grades of textile wastes (fly picker, motes, sweeps) was prepared on our cotton fiber blending equipment in the laboratory. The fibers were processed into a 16 ounce per square yard picker lap for ease of handling. The picker lap was separated into 10 inch widths and encased in gauze, again to facilitate handling on the processing equipment available to us. As a general rule individual samples for treatment weighed between 1/3 and 1/2 pound.

The treating solution (approximately 3 1/2 to 4 pounds) was made up to contain a calculated amount of solids of the treating chemical. The gauze encased picker lap was passed through the treating solution with some hand manipulation, then through a set of squeeze rolls, following which the sample weight was checked to ascertain the approximate wet add-on. The wetted sample was then repassed through the solution and through the squeeze rolls which had been adjusted to give a wet pickup of approximately 100 percent by weight to the exiting sample.

The wet samples were then dried (and in some cases cured) in a cabinet type oven. Where drying only was carried out, the samples were removed from the oven when their moisture content was between 10 and 12 percent by weight. The drying conditions seem to be somewhat critical. If high temperature air (over 220 ° F) was used, an undesirable amount of migration of the treating chemical to the surface of the picker lap usually resulted. As a consequence the interior of the sample was often deficient in flame resistance. All of the treated picker laps were air equilibrated for at least 24 hours before being garnetted.

Listed below are the results obtained when a series of runs were made to ascertain the effective lower limit of chemical add-on for a number of flame retardant treatments.

The first of this series was made using Pyrosan B. This borated amido polyphosphate met all of the criteria for effectiveness at all levels tested, that is, 5.4, 7.5, and 11.3 percent add-on, with one minor problem. Several of the samples had afterglow of short duration, probably due to the borax present. While we would like to have zero afterglow we consider the samples prepared from this product acceptable for most cushioning and mattress applications.

Pyrosan AN, a propyl ammonium phosphate, performed well at all of the levels tested, 5.3, 10.8, and 15.3 percent, and under all test conditions except mild leaching followed by drying at 158° F. for 16 hours. Under these conditions, the 5.3 percent add-on level sample failed by having a 12 second afterflame and 4-second afterglow and an excessive char length. At the 10.8- and 15.3-percent add-on levels, mild leaching did not reduce the flame retardancy below acceptable levels.

Shown below is a similar comparison of the effectiveness of Pyroset G2 and G2A (dicyandiamide derivatives) at different add-on levels.

Table 3.—Effect of add-on on flame retardancy

		Pyrosan B			Pyrosan AN		
Percent add-on 5.47.5 11.35.310.815.3							
70 °F—65 percent R.H.:							
Afterglow	sec.	3	0	0	0	0	0
Overall char	cm.	2	2	1	12	4	3
100° F—100 percent R.H.:							
Afterglow	sec.	1	1	1	0	0	0
Overall char	cm.	4	1	2	7	3	3
158° F—16 hr. (dry):							
Afterglow	sec.	2	2	0	0	0	0
Overall char	cm.	4	1	1	5	2	3
158° F—16 hr. (wet):							
Afterflame	sec.	0	0	0	12	0	1
Afterglow	sec.	2	1	0	4	0	0
Overall char	cm.	4	3	3	16	5	5

Table 4.—Effect of add-on on flame retardancy

		Pyroset G2			Pyroset G2A		
Percent add-on		5.6	8.1	11.3	5.4	8.2	11.2
70° F-65 percent R.H.:							
Afterglow	sec.	0	0	0	0	0	0
Overall char	cm.	7	6	4	7	6	5
100° F—100 percent R.H.:							
Afterglow	sec.	0	0	0	0	0	0
Overall char	cm.	10	6	3	4	4	3
158° F—16 hr. (dry):							
Afterglow	sec.	0	0	0	0	0	0
Overall char	cm.	15	6	3	8	8	4
158° F—16 hr. (wet):							
Afterflame	sec.	0	0	0	0	0	0
Afterglow	sec.	0	0	0	0	0	0
Overall char	cm.	6	4	3	9	5	4

Table 5.—Effect of add-on on flame retardancy

		Pyroset G			Tanotard F35		
Percent add-on		13.0	18.3	25.4	4.7	11.0	15.9
70° F—65 percent R.H.:							
Afterglow	sec.	0	0	0	0	0	0
Overall char	cm.	4	2	2	10	3	1
100° F—100 percent R.H.:							
Afterglow	sec.	0	0	0	0	0	0
Overall char	cm.	1	1	1	11	3	3
158° F—16 hr. (dry):							
Afterglow	sec.	0	0	0	0	0	0
Overall char	cm.	2	1	1	15	2	3
158° F—16 hr. (wet):							
Afterflame	sec.	0	0	0	3	0	0
Afterglow	sec.	0	0	0	0	0	0
Overall char	cm.	2	2	1	8	4	5

Table 6.—Effect of add-on on flame retardancy

		Caltac S			C904			
Percent add-on		4.9	7.9	10.4	3.1	5.3	8.8	11.4
70° F—65 percent R.H.:								
Afterglow	sec.	0	0	0	0	0	0	0
Overall char	cm.	F	15	16	F	7	1	1
100° F—100 percent R.H.:								
Afterglow	sec.	0	0	0	0	0	0	0
Overall char	cm.	F	F	F	F	8	2	2
158° F—16 hr. (dry):								
Afterglow	sec.	0	0	0	0	0	0	0
Overall char	cm.	F	F	12	F	8	3	2
158° F—16 hr. (wet):								
Afterflame	sec.	24	19	20	7	0	0	0
Afterglow	sec.	0	0	0	0	0	0	0
Overall char	cm.	F	10	13	8	4	3	3

NF = flashed to top

The Pyroset G2 performed well within acceptable limits when used at the 8.1- and 11.3-percent add-on levels under all test conditions. When this compound was used at the 5.3-percent level, although its performance was within acceptable limits, however the overall char length obtained after subjecting the sample to 158° F for 16 hours (dry) was 15 cm.—just under the 15.25 cm. length that had been more or less arbitrarily selected as the maximum permissible.

The Pyroset G2A showed acceptable performance at all three levels of add-on, 5.4, 8.2, and 11.2 percent, under all of the test conditions. Looking at the results we would be tempted to suggest that the 5.4 percent level might be a little low, and that products obtained with about 8.2 percent add-on would minimize the chance of rejection should plant controls slip up somewhere along the line.

Results obtained with Pyroset G and Tanotard F 35 are shown in the next table.

It should be noted at this point that the Pyroset G shown in this table the Pyroset G2 and the Pyroset G2A shown in the previous table, were experimental products at the time that they were evaluated for this application. They should not be considered the same as the product now being marketed under the trade name Pyroset G2.

Because we were unsure of the amount of chemical that would do the job, excessively high add-ons were used in these tests. From the performance we observed, it is obvious that a lower add-on than we selected would confer adequate flame retardancy on the products. The Pyroset G2 treated samples passed all requirements under all test conditions.

In the case of the Tanotard F 35, a mixture of organic and inorganic salts, it appears that the lowest add-on level of 4.7 percent was marginal in flame retardancy protection (15 cm. char length), particularly where the sample has been subjected to 158° F (dry) for 16 hours. There was some afterflame when the sample was subjected to 158° for 16 hours after soaking (wet). In this case, however, the char length was acceptable. At the 11- and 15.9-percent add-on levels, the samples were acceptable under all of the test conditions.

Table 7.—Effect of Add-on on Flame Retardancy

		Gaftex 281			Flamort TC			Resinox
Percent add-on		6.9	10.2	16.3	10.4	15.5	21.6	5.0
70° F—65 percent R.H.:								
Afterglow	sec.	0	0	0	0	0	0	0
Overall char	cm.	8	2	2	9	1	2	15
100° F—100 percent R.H.:								
Afterglow	sec.	0	0	0	0	0	0	0
Overall char	cm.	6	7	1	15	3	2	6
158° F—16 hr. (dry):								
Afterglow	sec.	0	0	0	0	0	0	—
Overall char	cm.	10	3	2	F	3	1	—
158° F—16 hr. (wet):								
Afterflame	sec.	11	4	0	5	6	0	4
Afterglow	sec.	0	0	0	0	0	0	0
Overall char	cm.	F	7	2	8	5	2	15

[7] **F = flashed to top**

Test results obtained when Caltac S, a borated urea formaldehyde condensate, and C 904, a urea phosphate, were used at various levels of add-on are shown in table 6.

For Caltec S at the 4.9-, 7.9-, and 10.4-percent levels, the treated products failed to pass the requirements for acceptability. They either flashed to the top of the sample, or exhibited afterflame or excessive char lengths.

In the case of C 904 it can be seen that the 3.1-percent add-on level was just not enough to confer an adequate degree of flame retardancy on the products. At this low level of add-on, the samples flashed to the top under most of the test conditions. However, when the add-on was increased to 5.3 percent, all of the samples successfully met the requirements under all of the test conditions. When the add-on was increased to 8.8 percent, there were small improvements in the char length under all of the test conditions. When the add-on was increased to 11.4 percent, the test results did not change significantly from those obtained at the 8.8 percent add-on level. The optimum add-on would therefore seem to fall between 5.3 and 8.8 percent for the C 904, and samples containing these amounts were acceptable in all respects for flame retardant cushion uses.

The effects of flame retardant add-on level for Gadtex 281, Flamort TC, and Resinox TS 6742 are given in table 7.

Flamort TC and Gaftex 281 are both mixtures of organic salts, and Resinox TS 6742 is a phenol derivative.

Both Gaftex 281 and Flamort TC were ineffective at the lowest levels of 6.9 and 10.4 percent, respectively. At the 10.2 percent add-on level, the Gaftex 281 gave acceptable flame retardancy; however, the performance after mild leaching, which showed an afterflame time of 4 seconds, is considered marginal. At the 16.3 percent level, this compound gave acceptable flame retardancy for all test conditions. Flamort TC gave acceptable performance at the 15.5 percent level except where the sample was subjected to mild leaching before testing. The 6-second afterflame under these conditions is considered unacceptable. At the 21.6-percent level samples exceeded the acceptable flame retardancy requirements under all test conditions. Some difficulty was experienced in the shelf life and water dilutability of the Resinox TS 6742, and it was only possible to obtain one sample at the 5-percent add-on level. The performance of this sample was marginal at best with a 1-second afterflame at 70 ° F., 65 percent RH, and a 15 cm. overall char length. Likewise the 4-second afterflame and the 15 cm. overall char length would be questionable. We plan to run additional tests with this product in the near future. One drawback, however, seems to be the phenolic odor that becomes evident when the product is heated to 158 ° and also when it is exposed to high relative humidity.

On the basis of the data presented so far, there seems to be every reason to believe that there are a number of commercially available chemical systems that offer promise of effective flame retardancy for cotton batting products. From our data, the add-on levels needed with several of these chemicals and their price make the production of acceptably flame retardant products possible at a nominal cost. The panel following this presentation will discuss with you the available commercial flame retardants and I am sure that they will want to talk about chemical cost themselves.

As I was discussing the flame retardancy properties of a number of the chemicals that we have evaluated, I called your attention to poor performance in several cases. The poorest performance was usually observed when the treated samples were subjected to 158 ° F. for 16 hours, either dry or after soaking with water.

Our efforts to improve the durability of the treatment under the above conditions have been in two directions, both of which could be expected not only to improve the durability of the flame retardant properties, but also to improve the resilience characteristics of the individual fibers within the batting array. These approaches involve the use of either a thermoplastic resin or a thermosetting resin. The function of these materials is to bond the flame retardant to the cotton more effectively and to make the flame retardant more difficult to remove by soaking or exposure to elevated temperature.

As we mentioned earlier in this presentation, care must be exercised in the selection of the thermoplastic resin to avoid the bonding together of the fibers which would increase fiber breakage during subsequent garnetting. A promising approach in this direction seems to be the use of truly water soluble ammonium salts of acrylic polymers. Even so, the add-on of such resins must be kept to a very low level if the fiber breakage is to be avoided.

The effect of thermoplastic resin by adding WR 1980-3, a water-soluble ammonia based acrylic resin, to a C 904 (urea phosphate) treating formulation are shown in table 8.

For comparison, we show two samples treated with only the C 904. You will note that at the 2.5-percent add-on level, the presence of the acrylic in the treating formulation reduced the char length except where the sample had been subjected to soaking followed by drying at 158° F. for 16 hours. In this case there was an insignificant increase in char length. You will observe that when the acrylic concentration was increased to 3.3 percent, some small improvement in the performance is obtained only under certain test conditions. However, after soaking and drying at 158° for 16 hours the sample had a 4 second afterglow and a 2 cm. greater char length than did the sample in the first column that did not contain the acrylic. While we have not yet obtained resilience performance data, we have every reason to believe that the presence of the acrylic resin should give the products better recovery from compressive loading.

The flame retardancy properties of cotton batting products, treated with various flame retardant chemicals, are altered when we include a thermosetting resin in the formation. The effects of thermosetting resins are shown in table 9.

Table 8.—Effect of thermoplastic resin on flame retardancy

Percent add-on C904		5.3	6.7	6.8	8.8
WR1980-3		—	2.3	3.3	2
70° F—65 percent R.H.:					
Afterglow	sec.	0	0	0	0
Overall char	cm.	7	3	4	1
100° F—100 percent R.H.					
Afterglow	sec.	0	0	0	0
Overall char	cm.	8	6	4	2
158° F—16 hrs. (dry):					
Afterglow	sec.	0	0	0	0
Overall char	cm.	8	3	3	3
158° F—16 hr. (wet):					
Afterflame	sec.	0	0	0	0
Afterglow	cm.	0	0	4	0
Overall char	cm.	4	5	6	3

Table 9.—Effect of thermosetting resin on flame retardancy

		Pyroset G			Caltac S				C 904
Percent add-on		12.4	13.0	7.0	7.0	7.9	6.6	6.6	5.3
Cyanabat CB		6.2		5.0				6.0	
Permafresh 183					5.0		6.0		
70° F—65 percent R.H.									
Overall char	cm.	2	4	3	1	15	12	3	7
100° F—100 percent R.H.									
Overall char	cm.	2	1	10	3	F	12	5	8
158° F—16 hr. (dry):									
Overall char	cm.	2	2	3	3	F	F	8	8
158° F—16 hr. (wet):									
Overall char	cm.	2	2	6	4	10	6	5	4

In the case of Pyroset G, a dicyandiamide derivative at the 12.4-percent add-on level, the addition of 6.2 percent of Cyanabat CB (a melamine resin) does not significantly change the flame retardancy properties of the products. As was mentioned earlier, we believe that the 12.4-percent add-on of the Pyroset G2 was far in excess of what was needed to confer adequate flame retardancy. We believe also that the presence of the thermosetting melamine will enhance the resilience of the individual fibers within a batting array, although we have not completed our tests to verify this.

The next three columns deal with Caltac S, a borated urea-formaldehyde condensate. You will recall that in table 6 we showed that this chemical was not too efficient in conferring flame retardancy on the batting products. In the first column we show that the addition of 7 percent Caltac S and 5-percent Cyanabat CB (a melamine) did improve the performance of the products, compared with the control shown in the third column which contained only the Caltac S. The samples did not do well when they were soaked and then dried at 158 ° F., having a 19-second afterflame, the same value as was obtained with the Caltac S alone. Otherwise they showed a considerable improvement in flame retardancy. Now, however, if we look at the middle column of data under the Caltac S, we note that there is a dramatic improvement in flame retardancy when we use a dimethyloldihydroxy ethyleneurea resin as the thermosetting component. With this thermosetting resin present, the samples demonstrated an acceptable degree of flame retardancy under all test conditions and would be acceptable for use in all cushioning applications.

In the last three columns, we show what happens when we include thermosetting resins with C 904. In these tests we used a chemical add-on of 6.6-percent C 904 and 6-percent of either melamine or dimethyloldihydroxy ethyleneurea. As a control we show in the last column a sample treated with 5.3 percent of C 904 only. In some cases there was very significant change in the flame retardancy over that shown for the control. Not all of these samples would be acceptable for cushioning applications. The sample containing the melamine is considerably better than the one containing the dimethyloldihydroxy ethyleneurea. The sample containing the melamine would be acceptable. The sample containing the dimethyloldihydroxy ethyleneurea would not. It will be noted that this is the opposite of our findings with Caltac S where the dimethyloldihydroxy ethyleneurea gave better flame retardancy than the melamine.

CONCLUSIONS

From the data presented, that there are a number of chemical systems that will confer a significant degree of flame retardancy on cotton batting. The economics of the treatment seem to be within reason, with the chemicals costing from 20¢ to 40¢ per pound, and add-ons needed between 6 and 10 percent by weight of the cotton fibers treated.

The chemical systems that we have evaluated so far seem also to meet the criteria of permanency, resistance to removal by elevated temperature, mild leaching, and high relative humidity, lack of physiological effect, and are relatively easy to apply from a water system.

Plans for future work:

Now, where do we go from here? A number of questions remain to be answered. First of all, we need guidelines that will define for us what level of flame retardancy will be necessary if the products are to comply with the intent of Congress. This is of course a very involved problem. It becomes necessary for the research team to establish on their own, or ascertain from other agencies, what properties to measure, how to measure them, and then how to interpret the results that have been obtained.

Let me give you a few examples. In cushioning applications which is more important—flame retardancy or rate of burning? Would a product that exhibited self extinguishing characteristics be satisfactory? Each of these characteristics would have to be measured in a different way using a different test procedure. How important is afterglow? Can we tolerate any afterglow?

Finally, how toxic are the byproducts from the combustion of the treated cotton batting? This would, of course, depend to a great extent on how the test procedure is set up—in a confined area, or under conditions where you have a 10 mile an hour breeze blowing over the sample.

It is our understanding that the U.S. Department of Health, Education and Welfare is making a survey to ascertain the precise causes of death in mattress and home fires. When this data become available it may shed some light on what the requirements for flame retardancy in cushioning applications might be. Meanwhile it would seem to the research team that the cotton batting industry should try to anticipate the implementation of the Flammable Textiles Act by initiating their own standards. Admittedly such standards will have to be modified as the state of the art improves. Such a move by the industry would be of great help to the research team in establishing goals for the present and for the foreseeable future.

There are other problems to be solved also. As I have pointed out, we have only examined in depth one technique (immersion) for the application of the chemicals to cotton fibers. We plan to investigate a number of other possible ways to apply the chemicals, anticipating that an inexpensive system to help even the smallest batting manufacturers can be found.

Appendix "A"
Manufacturers of Chemicals Used in this Work

Flame Retardants — Trade Name	Manufacturer
Pyrosan B Pyrosan AN	Laurel Products Corporation P. O. Box 1083, Charlotte, N. C. 28201
Pyroset G Pyroset G2A Pyroset G2	American Cyanamid Co. Bound Brook, N. J. 08805
Tanotard F 35	Chas. S. Tanner Co. P. O. Box 3867, Park Place, Greenville, S. C. 29608
Caltac S	Imperial Chemical Industries 55 Canal Street, Providence, R. I. 02901
C 904	Bendix Corporation Marshal Eclipse Division P. O. Box 238, Troy, N. Y. 12181
Gaftex 281	G.A.F. Corporation Textile Chemical Division P. O. Box 6037, Chattanooga, Tenn. 37401
Flamort TC	Flamort Chemical Co. 746 Natoma St., San Francisco, Calif. 94103
Resinox	Monsanto Company P. O. Box 2130, Springfield, Mass. 01101
Resins —	
WR 1980-3	Celanese Resins P. O. Box 8248, Louisville, Ky. 40208
Permafresh 183	Sun Chemical Corporation Wood River Junction, R. I. 02840
Cyanabat CB	American Cyanamid Co. Bound Brook, N. J. 08805

DISCUSSION

Question: If cotton fiber were treated for fire retardation at the ginning stage, what would be the effect on spinning or other physical properties?

Mr. Knoepfler: In general, any chemical treatment before the mechanical processing of cotton fibers into yarns and fabrics results in a reduction in the quality of the product. Stated another way, we could say that any time cotton fibers are pretreated before mechanical processing, the physical properties of the fibers are changed. This usually shows up as increased fiber breakage during opening, cleaning, carding, drawing, and spinning. Going one step further in the weaving, we find that yarns made from chemically treated rawstock usually abrade more easily on the loom and as a consequence more yarns are broken.

It was pointed out in my presentation today that the pretreatment of cotton fibers for flame retardancy almost invariably results in more fly—that is broken fibers—at the garnett. We hope that we will be able to minimize this problem in future research.

Question: What is the chemical cost per pound of dry fiber for the durable flame resistant finishes?

Mr. Knoepfler: First of all, let us distinguish between the different classes of flame retardants for cotton. Some of the treatments are durable to multiple launderings while still maintaining flame retardancy in the cotton product. These we call durable finishes. They are best represented by THPC (tetrakis (hydroxymethyl) phosphonium chloride), APO (tris (1-aziridinyl) phosphine oxide), and TBPP (tris (2-, 3-dibromopropyl) phosphate). These chemicals cost about $1 per pound and add-on of chemical to achieve acceptable flame retardancy is somewhere in the neighborhood of 10 percent by weight. This then means that for such treatments the chemical cost is about 10¢ per pound of cotton.

Now, let us look at what is called semidurable flame retardant finishes. These treatments confer flame retardancy for the life of the article as long as the article is not washed. Typical of this type of flame retardant are such chemicals as the urea-phosphates, borated amido polyphosphates, phosphorylated dicandiamide derivatives and the like. For cotton batting, these chemicals seem to be effective at about the 8-percent add-on level. They usually sell for 20¢ to 30¢ per pound, making the chemical cost for flame retardancy somewhere between 1.6¢ and 2.4¢ per pound of cotton treated.

PANEL DISCUSSION:—AVAILABLE FLAME RETARDANTS

PYROSET FLAME RETARDANT G-2

by

R. G. Weyker
American Cyanamid Co.
Bound Brook, N. J.

I welcome this opportunity to acquaint you with a product that imparts excellent flame retardant characteristics to cotton batting and, in so doing, detracts virtually not at all from other important features of batting such as hand, tensile strength, and resiliency. This product has been designated PYROSET Flame Retardant G-2, and some of its compositional features are:

	Percentage
Solids	55
Phosphorus	6.5
Nitrogen	11.8
Free formaldehyde	6
pH	3

The product is a clear aqueous solution that may be applied either by padding or spraying. As little as 5 to 6 percent of solids on the weight of batting has provided cotton batting that passes the General Motors (GM) tests. Table 1 shows some of the results obtained.

Three levels of PYROSET G-2 were applied and the treated batting was subjected to the four GM conditioning procedures before being tested.

As you can see, all levels of application gave zero afterflame, and afterglow was very slight in the case of two samples. There is a decrease in char length with increasing add-on, but even at the relatively low level of 5.4 percent solids, the main char length was less than 1 inch and the overall char less than 4 inches.

The treatments shown on this table were applied by soaking the raw stock in a solution of PYROSET G-2, after which the cotton was squeezed between rolls to a wet pickup of about 100 percent. In these instances the wet cotton was dried for 1½ hours at a temperature just below 200° F, but we have seen similar results when the drying was conducted at a temperature of 350°. After drying, the cotton was garnetted with very little fly generated during the operation. There was no dusting or flaking on subsequent flexing or handling, and the hand was virtually unchanged from that of untreated batting.

This flame retardant is priced at 15¢ per pound in bulk and 16¢ in truck load drums, f.o.b. Charlotte, N. C. Taking the drummed price, the add-ons shown here related to product costs of 3.3¢ per pound of cotton at the 11.2-percent level; 2.4¢ per pound at the 8.2-percent level, and 1.6¢ per pound at the 5.4-percent level.

PYROSET G-2 can also be coapplied with certain binders to provide flame retardant Cotton Flote. Table 2 show the results obtained via the General Motors tests for two applications made with different thermosetting binders. Sample A contains a glyoxal resin and Sample B used a melamine resin, both at 10

Table 1.—PYROSET flame retardant G-2. application on cotton batting

Flame tests	70° F. 65 pct. RH			100 pct. RH 3 days			158° F. 16 hr.			After water saturation			GM specifications
Pct. add-on	11.2	8.2	5.4	11.2	8.2	5.4	11.2	8.2	5.4	11.2	8.2	5.4	
Afterflame (sec)	0	0	0	0	0	0	0	0	0	0	0	0	Not over 5 sec.
Afterglow (sec)	0	0	0	0	3	4	0	0	0	0	0	0	Less than 1 min.
Overall char (in)	2.0	2.4	2.8	1.2	1.6	1.6	1.6	3.2	3.2	1.6	2.0	3.5	Not over 6 in.
Main char (in)	0.4	0.8	0.8	0.4	0.8	0.8	0.8	0.8	0.8	0.8	0.8	0.8	

Table 2.—PYROSET flame retardant G-2: application to Cotton Flote (sample A—glyoxal resin.

	70° F 65 pct RH		100 pct. RH - 3 days		158° F - 16 hr.		After water saturation		GM specifications
Sample	A	B	A	B	A	B	A	B	
Afterflame(sec.)	0	0	0	0	0	0	0	0	Not over 5 sec.
Afterglow(sec.)	15	6	12	10	16	16	14	15	Less than 1 in.
Overall char(in.)	3.5	2.8	3.2	4.0	4.3	3.5	5.9	5.1	Not over 6 in.
Main char(in.)	0.8	0.4	0.8	0.8	0.8	0.8	1.6	1.6	

percent resin solids. The thermoplastic binder is an acrylic, at 10 percent solids in both cases, and 10 percent of PYROSET Flame Retardant G-2 solids was coapplied with the other binders. The results are all within specifications, although in one or two cases char lengths approach the upper limit.

PYROSET Flaxe Retardant G-2 is a dimethylolated compound and as such can cross-link cellulose under the proper conditions. At modest levels of add-on, such as 5 to 8 percent solids, flame retardancy is obtained on raw cotton and on cotton batting, but when the add-on is increased to about 15 percent, PYROSET G-2 contributes to bond strength and higher tensile strength of the batting. In Cotton Flote, this effect could permit the use of less bonding agent and hence result in an overall cost reduction.

We have seen one instance, in fact, where raw stock was treated with PYROSET G-2 and an amine hydrochloride catalyst, after which it was dried and garnetted and then subjected to a cure at about 325° F. The batting showed tensile strength superior to that of regular garnetted cotton, and an increase in resilience as well. The amine hydrochloride catalyst is preferred in this application since it will not acQuire the "soggy" feel sometimes produced by high humidity.

PYROSET G-2 is not compatible with all of the thermoplastic latices considered appropriate for Cotton Flote. In general, compatibility is better when a glyoxal resin is used rather than a melamine resin.

In summary, PYROSET Flame Retardant G-2 can be used to provide a high degree of flame retardancy to cotton batting at a materials cost of about 2¢ per pound of cotton. The application procedure is simple and straightforward, and the treatment has no adverse effect on the cotton.

AVAILABLE FLAME RETARDANTS

by

J. F. Sturgeon
Bendix Corporation
Troy, N. Y.

Introduction and description of chemicals.—There are several flameproofing compounds presently being manufactured by the Bendix Corporation, Friction Materials Division, Troy, N. Y., for use by the textile industry.

1. *C-904* is a clear, water soluble, flame-retarding, urea complex designed for use in coating processes demanding stable fire-retardant finishes on cotton batting, rayon, paper, and other cellulosic materials. This thermo-reactive, water base system may be used by itself or in varying proportions with many thermosetting and/or thermoplastic resins to provide cellulosic articles with a wide variety of physical properties. The flameproofing characteristics may be controlled over a wide range depending on add-on and each individual manufacturing need and cost allowance.

2. *C-905* is a chemically reactive, thermoplastic, flame retarding emulsion. This water base system possesses the same coating versatility as C-904 and in some cases many distinct advantages over the latter, especially where wet strength, color, and fiber stiffness may be of primary importance. It is designed to provide the manufacture of various articles to be flameproofed with a one-coat system to replace several different resinous materials in his produce line.

3. *C-913* is a chemically reactive, thermoplastic, flame-retardant emulsion, chemically similar to C-905 and designed to improve: (1) the char length in flame-retardant Cotton Flote (both overall and main), (2) the percentage of heat set, and (3) the resistance to compressional loadings at 60 and 100 percent relative humidity.

In addition to the above materials, and since the last USDA interim report covering the period March 1967 to August 1967, two other flameproofing resins were developed: (1) C-922—a chemically reactive, thermoplastic, flame retardant emulsion. Although this resin was not tested by the USDA, we would possibly expect some improvements in physical performance over C-913 in the Cotton Flote process; and (2) C-926—a thermosetting, flameproofing resin designed for use in automotive air filter. This material could possibly replace, either partially or totally other flameproofing materials and/or thermosetting resins in Cotton Flote.

Use of flameproofing resins in Cotton Flote Based on USDA's work (interim progress reports XII and XIII covering the period from September 1966 through August 1967) and experimentation at the Bendix Chemical Products Laboratory, we can generally conclude that in a Cotton Flote formulation, 50-percent C-905, 35- to 40-percent C-904, or 50-percent C-913 can give satisfactory flameproofing without afterglow. This is equivalent to a solids add-on of 10-percent C-905, 8-percent C-904, and 10-percent C-913 when the solutions are applied at 100-percent wet add-on. As little as a 5 weight-percent of C-913 can be used successfully when incorporated with a flameproof acrylic. The leaching resistance is excellent for all aforementioned materials when extracted with water, as described in Engineering Section Test Procedure No. 30-27 revised 2/3/67, Fisher Body Division, General Motors Corporation. The flame resistance after exposure to 100 percent relative humidity at 100 ° F. for 3 days, in accordance with the aforementioned Fisher Body Division test procedure, is excellent for all three resins when applied at the above add-on.

In the separate application of fiber pretreatment, C-904 offers good possibilities of achieving the desired characteristics in a cotton product. One outstanding property is that in the pretreatment of fibers with C-904, adequate flameproofing is achieved by only drying the cotton article. A cure is not necessary. A solids add-on of 8 percent is adequate.

In the course of experimental development of low cost flameproofing compounds, several urea ammonium phosphate salt and/or phosphoric acid mixtures were prepared. These materials were compared to C-904 in a study of fiber performance on coated automotive air filter paper. In general, the salt treatments greatly reduced the fibers' tensile strength, tear strength, and water resistance. The cohesiveness and structural stability were also poor, and the flame resistance is lost after a mild leach in water. We would expect similar results when the salt treatments are employed to flameproof Cotton Flote.

Summary.—The problems of developing an effective flame retardant for cotton batting are complicated by many factors. The selection of a flameproofing system should be based on the following requirements:

1. It should be a low cost treatment in relation to product cost.
2. It should be a water system.
3. It must be effective at low add-on levels.
4. It must be glow resistant and must not be removed by mild leachings.
5. It must not affect the air permeability of the array or the resilience under load.

Again, when considering that a range of cotton products will be manufactured with different resiliencies, densities, structural stabilities, etc., the choice of the optimum coating system (flameproofing resin, thermoplastic, and/or thermosetting resin) will depend on performance requirements for the specific end use.

For high performance Flameproofed Cotton Flote, C-904 combined with an acrylic-vinyl chloride copolymer and a methylated methylol melamine works very satisfactorily. In addition, C-904 is recommended for pretreatment of fibers.

In applications where a lesser degree of performance is required, a single chemical system such as C-905, C-913, or C-922 should prove adequate.

The cost of the above flameproofing resins are as follows:

	C-904 33-pct. solids	C-905 36-pct. solids	C-913 36-pct. solids
Less truckload (drums)	$0.15	$0.19	$0.19 per lb.
Truckload (drums)	.14	.18	.18 per lb.
Tank truck	.13	.17	.17 per lb.

	C-922 30-pct. solids	C-927 55-pct. solids
Less truckload (drums)	$0.16	$0.39 per lb.
Truckload (drums)	.15	.38 per lb.
Tank truck	.14	.36 per lb.

A FIRE RETARDANT FOR COTTON BATTING

by

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Laurel Products Corporation
Charlotte, N. C.

The long history of man's frustrating and largely unsatisfactory attempts to develop suitable fire retardants for clothing and other cellulosic textile materials, in the continuing battle against fire, is well known. Thousands of formulations, combinations, mixtures of borates, phosphates, sulphates, sulfamates, etc., have been made; coatings, chemical modification of the fiber and resin treatments have been tried; hundreds of patents issued, a great deal of experimenting done; a vast data of testing information gathered; and a variety of test methods developed, nearly all of which are empirical. Beginning with World War II, serious efforts have been made to examine the chemical and physical phenomena involved in the inhibition, by finishing processes, of the pyrolytic degradation and combustion of cotton with flaming and afterglow. Much of the recent work has been done here in New Orleans.

Laurel, after much of the aforementioned fumbling and largely by empirical methods, has settled on two main types of fire retardants:

1. Boro-phosphate complexes
2. Ammonium salts of the esters of alkyl phosphoric acids.

The first type would seem to be more interesting to batting manufacturers, particularly one marketed under the name of Pyrosan B. When properly formulated with suitable additives borax-phosphoric acid products are heavy viscous colorless liquids. They are water soluble, nontoxic, virtually odor free, store well, can be sprayed or applied from water baths, do not produce extremely noxious fumes when materials to which they are applied are exposed to flame, and are low in cost. The add-on for effective flame- and glow-resistance averages about 10 percent, depending on fabric and yarn construction or fiber form. For many years they have been applied to cotton sewing thread, yarns for insulation, cotton flannels, toy plush, cotton and rayon drapery and upholstery fabrics, velours, mattress ticking, walk off mats, disposable fabrics, and other cellulosic textile materials.

When correctly applied and dried at reasonable temperatures, hand, strength, and most colors are not greatly affected, nor are whites yellowed. The flame retardancy of cellulosic materials does not appear to undergo significant change on storage or exposure to light under normal use conditions. Borophosphate complexes flameproof most cotton materials as well as anything now offered as long as they are not washed—if the treated fabric is put into water most of the fire retardancy is lost. Softening agents stable to high concentrations of electrolytes, particularly some of the polyethylene glycol esters, may be used in conjunction with these fire retardants to improve lubricity and softness; nonionic surfactants may be used to increase wetting speed and penetration.

The exact chemical constitution of preparations such as Pyrosan B would be difficult to determine. They have been improved largely by experimentation, by trial and error; and produced often for specific applications. When washed, treated materials do show some small degree of residual flame resistance and there is some evidence of phosphorylation of the cellulose. Their chemical nature satisfies modern theoretical ideas concerning flameproofing and glowproofing.

The present uses being made of borophosphate complexes suggest their suitability for flameproofing cotton batting. Treatment could be by immersion in a solution of the fire retardant or by spraying. Application is the business of the panel to follow, but it is apparent that almost any type of raw stock bleaching or dyeing equipment, centrifuging, or continuous processes adapted to short staple fiber could be used. All that is required is to saturate the stock with the solution of the fire retardant at room temperature. Based on a 10-percent add-on, pickup, and the average current price of boro-phosphate complexes, the chemical cost would be about 2¢ per pound in a process where excess bath liquor was recovered. Flame resistance and glow resistance tests made in Charlotte, and by Mr. Knoepfler at the Laboratory here, indicate that 10 percent add-on would impart satisfactory fire retardancy to cotton batting —no afterflaming, no afterglow.

Improvement of fastness to washing occurs to everyone working with fire retardants of this type and many attempts to improve fastness have been made. Although there has been some progress, our efforts to bind the various effective water-soluble products by resin treatment and the like have met with indifferent success. Mr. Knoepfler advises that there has been some progress along these lines in Cotton Flote formulations. Process development and proper formulation of these inexpensive materials may eventually result in obtaining a degree of durability to washing and dry cleaning satisfactory for many end uses. Continued efforts along these lines would appear worthwhile.

Finally a comment about fire retardants in general, and testing. Virtually no one applies fire retardants voluntarily to a fabric as a plus or as a promotion. They are nearly always used by the textile manufacturer under duress. Realism forces the acknowledgment that there are really no completely satisfactory fire retardants for cellulosic textiles from the standpoints of esthetics, ease of application, and cost. Experience shows that process and formulation should be specific for the particular product and its end use; marquisette is not duck; loose fluffy cotton decorations are not compacted felts. This in turn requires realistic standards of performance; to demand what cannot be accomplished is useless. Certainly research in the development, application, and testing of fire retardants on the part of chemical companies, the textile industry, the associations connected with it, and the government, should continue and expand. Concerning testing, a few—just a few —of the many standards of performance and tests proposed by Federal and State governments, associations, and corporations are given below. One test will probably never suit all end uses, but it seems that in striving for perfection the efforts to develop tests sometimes become bogged down in the investigation of detail. All this may be necessary, but the ordinary match seems a pretty good tool. Although not a very scientific instrument, it often tells you what you want to know.

Rawstock Treatment

Sample No. Flame retardant Pct. Add-on	339106 Pyrosan B 5.4	339107 Pyrosan B 7.5	339108 Pyrosan B 11.3
Flame Retardant Tests			
70° F, 65 pct. RH:			
1) Afterflame (sec.)	0.0	0.0	0.0
2) Afterglow (sec.)	3.0	0.0	0.0
3) Overall char (cm.)	2.0	2.0	1.0
4) Main char (cm.)	1.0	1.0	1.0
100 pct. RH -3 days:			
1) Afterflame (sec.)	0.0	0.0	0.0
2) Afterglow (sec.)	0.0	1.0	1.0
3) Overall char (cm.)	4.0	1.0	1.5
4) Main char (cm.)	1.0	1.0	1.0
158° F - 16 hr.:			
1) Afterflame (sec.)	0.0	0.0	0.0
2) Afterglow (sec.)	2.0	2.0	0.0
3) Overall char (cm.)	3.5	1.0	1.0
4) Main char (cm.)	1.0	1.0	1.0
After Water Saturation:			
1) Afterflame (sec.)	0.0	0.0	0.0
2) Afterglow (sec.)	2.0	1.0	0.0
3) Overall char (cm.)	3.5	3.0	3.0
4) Main char (cm.)	1.0	1.0	1.0

Cotton Flote Physical Properties

Sample No.	339042	339043	339044
Pct. solids	20	20	20
Resin	Cyanabatt CB	PF 183	Cyanabatt CB
pct.	40	37.5	37.5
Latex	HA-20	Geon 450 x 167	Geon 450 x 167
pct.	45	37.5	37.5
Flame retardant	Pyrosan B	Pyrosan B	Pyrosan B
pct	15	25	25
Catalyst	$MgCl_2$	$ZnCl_2$	$MgCl_2$
Density (#/ft^3)	3.1	1.95	2.2
60 pct. RH 0'	88.4	86.0	89.0
Pct. Recovery 2' 4'	92.3	87.5	93.7
	92.3	89.0	95.3
100 pct. RH 0'	76.9	62.5	65.0
Pct. recovery 2' 4'	80.6	65.6	71.5
	80.6	65.6	73.0
Pct. compression	58.6	68.2	61.6

Cotton Flote Flame Tests

Sample No.	339042	339043	339044
70° F 65 pct. RH:			
1) Afterflame (sec.)	0.0	0.0	0.0
2) Afterglow (sec.)	0.0	0.0	0.0
3) Overall char (cm.)	2.5	3.5	2.5
4) Main char (cm.)	1.0	1.0	1.0
100 pct. RH-3 days:			
1) Afterflame (sec.)	0.0	0.0	0.0
2) Afterglow (sec.)	0.0	0.0	0.0
3) Overall char (cm.)	2.5	4.5	2.0
4) Main char (cm.)	1.0	1.0	1.0
158° F.- 16 hr.:			
1) Afterflame (sec.)	0.0	0.0	0.0
2) Afterglow (sec.)	0.0	0.0	0.0
3) Overall char (cm.)	3.5	3.0	2.0
4) Main char (cm.)	1.0	1.0	1.0
After water saturation:			
1) Afterflame (sec.)	0.0	0.0	0.0
2) Afterglow (sec.)	0.0	0.0	0.0
3) Overall char (cm.)	3.5	2.0	2.5
4) Main char (cm.)	1.0	1.0	1.0

TEST METHOD
MATERIAL DEVELOPMENT AND TESTING LABORATORY

No. 30-27

Fisher Body Division
General Motors Corporation
Research and Development Activity

Page No. 1

Issued: July 20, 1966

Revised: 2-2-67, 8-18-67, 9-19-67, 11-13-67

FIRE RESISTANCE (MATCH METHOD)

Application

This procedure is used to determine the fire resistance of materials commonly used in the interior of automobiles.

Apparatus Required

1. Wooden Matches — approximately 15 seconds burning time. We use 12-second flame from a bunsen burner per AATCC procedure.
2. Clamp
3. Stand

Procedure

1. Cut 8 test specimens 1" x 12" by the thickness of the part. If the part thickness exceeds ½", the test specimen shall be cut down to a thickness of ½" and will include the primary surface of the part. In the case of directional materials 8 specimens each shall be cut in both the warp and filling directions (machine direction and across machine direction). A mark shall be made in the middle of the 12" length at each specimen.
2. Two (2) specimens in each direction (warp and filling) shall be tested under each of the following conditions:
   - (a) Air circulating oven at 158 °F. for a minimum of 16 hours.
   - (b) 100 Pct. R.H., 100° F for 72 hours and dried in an air circulating oven at 158 °F. for a minimum of 16 hours.
   - (c) Saturation — Specimen is made to absorb small amounts of water until completely saturated. No water should be added in excess of the amount the specimen is able to absorb. The sample is then tried in an air circulating oven at 158°F. for a minimum of 16 hours.
   - (d) 65 Pct. R.H., 70 °F. for a minimum of 16 hours.

NOTE: To facilitate handling, samples may be taken from the oven and placed in a polyethylene bag or wrapping. Samples may remain in the polyethylene a mamimum of 1 hour. Allow specimens to cool to room temperature before proceeding with the test.

3. The specimen is suspended vertically lengthwise from the clamp.
4. The match is lit and used to ignite the bottom of the 1" edge of the specimen. The specimen is exposed to the flame of the match until the match is consumed (15 seconds).
5. If the flame is extinguished in 5 seconds after the match is consumed and the specimen has not burned past the center mark, then the material is considered satisfactory for fire resistance.
6. If any of the specimens are burned beyond the center mark, the material is unsatisfactory. If any of the specimens afterglows and/or smolders for one (1) minute after flame has gone out the specimen is unsatisfactory.

APPROVED BY: J. G. HAVILAND

Revised:

2-3-67	To include afterglow or smoldering in Item 6.
8-18-67	To up-date test method.
9-19-67	Added: 5 second duration of flame (Par. 5).
11-13-67	Item 1 — Thickness requirements added.

REACTIVE FIRE RETARDANT TEXTILE FINISH

by

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Hooker's background on fire retardant resin systems for woven piece goods is submitted as a possible means of making cotton batting durably fire-retardant.

I would like to be in a position to demonstrate mill processed cotton batting using the THPC resin systems, however, to date this has not been accomplished. Perhaps the best approach is to show you this sample of a 100-percent cotton thermo-blanket—a very loosely woven item finding wide acceptance in institutions. This sample is treated and represents what is commercially being done in both continuous and batch processing.

The THPC resin systems offer a number of advantages which are demonstrated by this sample.

1. Although there is 20 percent resin fixed to the fabric, the hand remains very pleasant and is almost indistinguishable from the untreated material.
2. The material is fire retardant (demonstrated by casual match test), withstanding a severe vertical char test, and afterglow is eliminated.
3. The fire-retardant system is very durable to water leaching, again as evidenced by this sample which has been through 20 washes.
4. Properties such as tensile- and tear-strength are essentially unaffected in this sample because of its high absorbency and the loose weave.
5. The products of combustion generated while the fabric is exposed to flame, although objectionable, have been evaluated both here at SURDD and by the Military at Wright-Patterson Air Force Base, and have been found to be no more toxic than the products of combustion of untreated cotton. Because of the self extinguishing nature of the fabric, the products of combustion are negligible.
6. Although the system involves a resin application, the moisture regain of the fibers is little affected. This system also imparts durable antibacterial properties and a degree of mildew and rot resistance.

From the foregoing it might appear that we have the ideal solution for fire-retardant cotton batting. However, there are some major obstacles to making this a commercial success for batting. One of these obstacles is processing. The system was designed for application to woven piece goods on typical finishing range equipment. By following the process as it applies to woven goods you may judge whether or not it would have application in your own processing procedure.

Fabric preparation.—The woven cotton or rayon fabric is scoured and desized to afford the greatest degree of absorbency. This is especially important in fabrics of tight construction where the resin system may adversely affect the tear strength and hand. I think it is safe to assume that this would not be a critical concern in treating cotton batting. We have treated some loose weave fabrics in the greige state without adversely affecting the tear strength or hand.

Pad.—Rather than go into the chemistry of the resin system I'll just mention that it does incorporate tetrakis (hydroxymethyl) phosphonium chloride, or using our trademark THPC ®. The piece goods are processed through the pad bath containing the THPC resin system and through a tight squeeze roll setting to obtain about an 80 percent wet pickup. There is substantial leeway in varying the pad bath concentration and the percentage of wet pickup. The prime concern is to end with approximately a 20-percent resin add-on after the water in the resin system has been driven off and the chemical reaction has taken place. I should point out that the resin level of 20 percent applies to woven goods processed to pass the vertical char test.

Dry.—After it comes out of the padder, the fabric is dried in either a tenter frame or roller oven, driving off the water from the resin system.

Cure.—The fabric is fed into the curing oven, which again is normally a roller oven, and is heated to approximately 300 ° F. for 2 minutes. This is where the resins react and are fixed to the fabric in a manner that will withstand water leaching and hot alkaline wash cycles.

Wash.—After the fabric comes out of the curing oven, some unreacted chemicals remain that pose an odor and irritant problem, and necessitate the fabric being washed, usually in a sodium perborate and soap soda ash series of wash boxes.

Dry.—The fabric is then dried and ready for use. Summarizing the process steps, I would suggest that the areas of greatest concern might be with the need to scour and afterwash.

Another obstacle to making this a commercial success for batting is cost. To date, our work has been concentrated in the woven goods field, where the "raw material" or prepared fabric has a substantially higher value than cotton batting. The THPC resin systems will add in the way of chemical costs from 15¢ to 20¢ per pound to woven goods with resin levels aimed at passing the vertical char test. Depending on the type of fire test cotton batting would undergo, the resin level and therefore the chemical cost may be substantially lower than those required for woven goods.

I think it's appropriate to mention other related areas where the THPC resin systems are effective. The systems are being applied commercially to mattress ticking, resulting in good durable fire retardant properties. Test mattresses covered with this ticking, and these mattresses included cotton batting, latex foam rubber, and urethane foam fill, were covered in a normal manner with treated ticking and exposed to a series of fire tests. Incidentally, the ticking was exposed to a number of wash cycles prior to making up the mattresses. These tests used glowing cigarettes and cigars (held in such a manner that the glowing end was always leaning on the ticking and periodically inhaled to maintain the glow), an ignited book of matches, glowing particles of charcoal to simulate pipe tobacco, a small woodfire, and an oil soaked rag. In all instances, the treated ticking offered sufficient protection to prevent the fill, whether it be cotton batting, latex foam rubber, or urethane foam, from igniting. This test was also successful when untreated bed linens covered the mattress ticking and when there was substantial movement of air over the mattress. In each control test, using untreated ticking, a fire was propagated in the fill.

Another item of related interest is upholstery. Airline upholstery fabric is being treated commercially with THPC resin systems. The flammability standard is usually a vertical or horizontal flame spread but, in view of the weight of this fabric, it is safe to assume that it would perform more effectively than ticking as a cover for cotton batting.

On an experimental basis, these systems have been used to treat the knit goods that are eventually vinyl coated for automotive upholstery.

Before closing, I would like to acknowledge the very substantial contribution the U.S. Department of Agriculture and, in particular, the Southern Utilization Research and Development Division, has contributed in the area of "Tetrakis" chemistry. This exceedingly useful chemical was born in 1921 when A. Hoffman reacted phosphine, formaldehyde, and hydrogen chloride to get tetrakis (hydroxymethyl)-phosphonium chloride. However, the chemical did not approach manhood until 1952 when the USDA applied for patents covering the application of Tetrakis to textiles to make them fire retardant. This was the real beginning, the point where the textile industry could start to benefit and we can thank our hosts for their efforts on our behalf.

BORATES FOR USE IN FIRE RETARDANCY

by

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INTRODUCTION

The use of borax, boric acid, and other borates to flameproof a variety of substrates has been known and practiced since the days of Gay Lussac, who, coincidentally, discovered the element boron in 1808. Because of the ready water solubility of most boron-containing salts, recent research on their use has been for the most part restricted to materials that are not subjected to water leaching, as by laundering or exposure to weather. For this reason, most of the research on the use of borates in fire retardancy has involved wood or wood products used in protected environments. This work has been highly successful and has led to the development of numerous fire retardant products at minimal cost. Concepts arising from the program should apply analogously to related cellulose products like cotton batting.

FR-28, an improved sodium borate fire retardant. One such concept was the development of a borate composition specifically adapted to use in fire retardancy. Most of us are familiar with borax and with boric acid (table 1). Borax is an outstanding flame suppressant at low add-on levels. It imparts little afterglow suppression, however, and its solutions are somewhat on the basic side of neutral. Boric acid, on the other hand, very effective in supressing afterglow or smoldering, is less efficient as a flame suppressant. Values of pH for solutions of boric acid are, of course, on the lower side of neutral. The well-known 7:3 borax/boric acid mixture, which has been recommended by various agencies for use in fabrics and paper, combines desirable features of both constituents. This combination is effective for reducing both flame propagation and afterglow, and does so at lower add-on levels than almost any other combination of inorganic salts.

A greatly improved composition and one that has yet to be fully exploited in textiles is the formula shown at the bottom of table 1. This material, designated FR-28, is a specially synthesized borate fire retardant composition that has several advantages over previous formulations.

1. It has a fixed composition and cannot segregate as mixtures sometimes do.
2. It has high water solubility and a fast solution rate.
3. Its water solutions exhibit pH values in the 7.8-8.5 range.
4. It is a highly concentrated source of boric oxide.
5. It is amorphous and therefore gives better "hand" in textiles and paper than crystalline products.
6. It is highly compatible with latexes of various types.
7. It retains the inherent low cost of borates.

Table 1.—Properties of various sodium borates

Boric Acid	pH/Soln. Conc. Pct.	B_20_3 (Wt. pct.)	Cost (Cents/lb.)
H_3BO_3	3.6/5	56.3	5.0 — 6.0
$B_20_3 \cdot 3/2\ H_20$	5.1/1		
Borax	9.2/10	36.5	2.0 — 3.0
$Na_2B_40_7 \cdot 10H_20$	9.1/5		
$Na_20 \cdot 2B_20_3 \cdot 10H_20$			
Borax/Boric Acid, 7/3	8.00/15	42.3	3.0 — 4.0
$Na_20 \cdot 3 \cdot 3B_20_3 \cdot 14H_20$	8.3/5		
FR-28	7.8/15	66.0	7.0 — 8.0
$Na_2B_80_{13} \cdot 4\ H_20$	8.2/5		
$Na_20 \cdot 4B_20_3 \cdot 4H_20$			

Research at U.S. BORAX has developed techniques and process parameters for industrial applications of FR-28, and this activity is continuing. The first application of the material was in fire retardant latex paints. A number of highly satisfactory formulations were evolved and are in use today. Acoustical tile was a subsequent area for application of FR-28. Wood fiber tiles impregnated with this material can pass the most stringent rating (Class I) in the Underwriter's 25-foot tunnel test. Physical properties of the tile are not adversely affected. Volatility of FR-28 is essentially nil, even during drying operations where temperatures up to 350 ° F. are encountered.

Work is now in progress at U.S. BORAX and elsewhere to broaden the application of FR-28 to other cellulosic materials. Particleboard, strawboard, plywood, and lumber have all been impregnated with FR-28 under various conditions to achieve high degree of fire retardancy. A new micronized version of FR-28 has been developed for use in plastics. Of direct interest to this conference is the fact that FR-28 has been successfully evaluated as a part of fire retarding systems for Cotton Flote.

A new zinc borate.—Zinc borates also have been known for some time in the field of fire retardancy. Some familiar variations of what are called "zinc borates" are shown in table 2. A new zinc borate now under development at U.S. BORAX, designated XPI-158, is also shown in this table. A similar approach to that which guided the development of FR-28R has been followed, namely the development of a new boric oxide-based system with properties specially tailored for use in fire-retardant products. The new compound has the composition

$$2Zn0 \cdot 3B_20_3 \cdot 3 \cdot 5H\text{-}_20$$

and thereby provides higher boron content, relatively low water content plus all of the advantages normally obtained with true compounds in contrast to loose mixtures or coprecipitates.

Table 2.—Zinc borates

$Zn0\ B_20_3.2H_20$	(112)
$2Zn0 \cdot 3B_20_3 \cdot 9H\ 0$	(239)
$2Zn0 \cdot 3B_20_3 \cdot 5H_20$	(235)
$2Zn0 \cdot 3B_20_3 \cdot 3 \cdot 5H_20$	(2335), XPI-158

The uses for this compound follow essentially the same pattern as those for the other zinc borates, but with superior performance. For the most part it is used as an economical partial replacement for antimony oxide in fire retardant plastics. Thus far it has been found effective on this basis in polyvinyl chloride polyesters and chlorinated polyesters. In certain chlorinated polyesters, XPI-158 is highly effective as a total replacement for antimony oxide. Water evolution from XPI-158 takes place at higher temperatures than for any other zinc borate. This property, coupled with low initial water content, provides the formulator with a whole range of processing and use conditions and helps minimize deterioration of physical properties.

Apparently zinc borate has not been studied extensively in cotton batting, although its application in formulations for fire resistant cotton fabrics for military use has been well documented. This compound could be potentially effective either as an additive to the batting itself or to the resins used in the fabrication process.

PANEL DISCUSSION: "Application of Chemicals to Cotton Fibers for the Production of Flame Retardant Batting"

APPLICATION OF CHEMICALS TO COTTON FIBERS FOR FIRE-RESISTANT BATTING

by

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Proctor and Schwartz, Inc.
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The present topic is the application of chemicals to cotton fibers for fire-resistant batting. Pending development of firm product specifications, it seems fair to say that the state of the art is a state of indecision. The basic questions which need to be answered before any process approach can be considered firm are:

(1) What are the product specifications?
(2) What costs can be tolerated?
(3) What are the characteristics of the materials in process? and
(4) What are the necessary process conditions?

Given answers to these questions, we could offer a treating range within a few weeks.

The probable key question is tolerable cost. By this I mean that legislating requirements for a fire resistant product does not say that the market place will accept higher costs for an otherwise equivalent product. In the present era of intense competition and broad technical competence, any significant increase in price levels is an open invitation for penetration by alternate materials, which may offer other active benefits in addition to the passive benefit of reduced fire hazard. I don't want to belabor this point, but I do want to suggest that this may well be the controlling factor in determining a preferred approach to treatment of cotton. I believe it is highly probable that properly designed fire-resistant treatment for cotton will result in corollary benefits which will largely or fully offset added costs. I think the possibilities for such secondary benefits will become clear as I outline our present position on chemical application techniques.

I'd first like to consider some of the basic factors in fiber processing that control the effective application of chemicals to raw cotton. To begin, raw cotton fibers are highly resistant to wetting, due to natural oils in the fibers and to air trapped in the densely packed clumps of very fine fibers. Conversely, all of our work to date indicates that thorough, uniform saturation of these fibers is necessary to avoid highly flammable, untreated patches or streaks of fiber in the final batting. Because of this difficulty in wetting, simple immersion treatments, as in a scouring bowl, have not given adequate distribution of chemicals. Although we cannot say that some acceptable means of immersion treatment cannot de developed, we have not pursued this route because of the low evaporative rates and consequent high investment cost in the subsequent drying operation, due to poor air penetration into the dense clumps of fiber.

A possible alternative is to open the fiber fully and to spray the lightweight web just prior to lapping, as in the Cotton Flote process. However, this would also lead to high investment costs if used solely for the application of fire-resistant chemicals. Additionally, if such a system were adapted to a central, high capacity treating operation, excessive work on the fiber would lead to high fiber breakage and excessive fly when the fiber is reworked to form the finished batting.

Our work to date has led to an alternate approach that minimizes investment and yet eliminates the principal problems just mentioned. This process is based on handling the fiber in a convenient batt form throughout the operation. However, the initial batt is formed from the cleaned fiber by a very coarse high capacity garnetting operation that performs the minimum work necessary to open the dense clumps. This batt is then carried through a special conveyor system, where it is wet out with solution by a coarse flooding spray system that forces solution through the batt. Excess solution is wrung out by pressure rolls and the batt is then opened prior to drying by a novel, pneumatic wet stock opening system. This permits uniformly high velocity through circulation of air in the drying operation, providing high capacity and minimum cost.

We have installed a pilot model 2 feet wide of this system and are in the process of ironing out some minor mechanical bugs. We have progressed far enough to confirm that we have the basis for a feasible commercial system, provided the product requirements, the chemical systems, and the operating conditions are reasonably close to those indicated by present published information. We have been and still are proceeding cautiously, however, because any significant shift in these assumed requirements, for example, might radically alter the mechanical requirements of the system.

Accepting the present state of the art as reasonably representative, we have made preliminary estimates of the costs of a 6,000-lb/hr bulk treating system. Purchased equipment costs would be $150 to $175,000 and operating costs would be 1/2¢ to 3/4¢ per pound. The system could be scaled up or down from about 500 to about 12,000 lb/hr without significantly altering the operating cost, provided efficient labor distribution could be maintained at lower volumes. This system assumes the use of a permanent, or at least resistant, chemical system and provides several minutes of holdup time for curing.

Operation of the system with urea-phosphate type chemicals has produced a uniformly fire resistant batt, with no afterflame or afterglow, and with phosphate levels of 4 to 5 percent and chemical costs of about 0.7¢ lb. The fire resistance is retained after mild leaching, prolonged storage, or exposure to high humidity. In some cases, the treated material has garnetted better than raw cotton and with less fly, provided the moisture content has been readjusted to reasonably high levels after curing. The system, as now projected, would not be much more complex than a conventional cloth scouring and drying range, and should be reliable and simple to maintain.

A final point is the one with which I started—the question of possible secondary benefits. Some chemical systems have shown significant improvement in properties, requiring 10 to 15 percent less cotton to give equivalent loft and compression resistance, with superior resilient recovery. Unfortunately these are not necessarily the same systems that give the best fire resistance protection. Nevertheless, the indication is that a properly compounded system could provide many of the benefits of Cotton Flote. It is quite possible, for example, that a compatible resin system could be incorporated into the treating solution, and the curing section relocated after the final garnetting and lapping operation, rather than being a part of the bulk system. This would provide completely uniform resin distribution. In this case, of course, the curing oven could be scaled to the capacity of the line. Since there is no drying load, this curing unit could be quite compact and inexpensive.

I would like to leave you with the thought that the possibilities for compensating savings in raw materials, or for a much improved product, are most realistic as well as probably necessary to maintain a competitive position. As one final fillip, I might point out that the same saturation, drying, and curing system works well for finished felts and cloth structures. This might permit a mattress manufacturer to buy and treat his own fabric in addition to the cotton padding.

VARIOUS METHODS FOR FIRE RETARDING COTTON BATTING

by

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Monsanto Chemical Company
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Monsanto Company is primarily interested in the chemicals used to fire-retard cotton products as opposed to the processing equipment aspect of this industry. However, we have developed information that pertains to the choice of equipment required to manufacture fire-retardant batting.

I will limit this discussion to those chemicals that will provide nonpermanent to semipermanent fire retardancy. These chemicals include the ammonium phosphates and modifications of them. Nonpermanent fire retardants are those that can be leached out, washed out, or physically removed from the substrate over a period of time. Semipermanent treatments are those that will withstand a certain amount of leaching, washing, or handling for some longer period of time.

We at Monsanto believe that under certain conditions nonpermanent to semipermanent fire retardant treatments will be preferred for both economic and performance reasons.

I will discuss the methods of fire retardant applications shown below.

Methods of fire retardant application:

1. Immersion or padding
2. Spraying
3. Roller coating

Perhaps the simplest technique for applying soluble fire retardants is to totally immerse the product in a fire retardant solution and then remove the excess moisture using squeeze rolls and a drying over. This technique can probably be used only on the finished batt, and will therefore mean the addition of padding and drying equipment at the end of the batting line.

The following procedure can be used to treat batting by this method, using for example monoammonium phosphate as the fire retardant.

Immersion or padding treatment:

1. Prepare 15 percent solution of monoammonium phosphate
2. Immerse the batt until thoroughly wetted
3. Pass the treated batt through squeeze rolls
4. Dry at a temperature not to exceed 270° F
5. Dry to the *maximum allowable* moisture content

In step 3 above, 100-percent wet weight add-on will be required after the nip in order to retain sufficient chemical. Additionally, we have found that fire retardant substantivity and durability to moisture leaching is improved with multiple passes through the nip.

Let us also explore the last two steps in depth. A previous study has shown that:

1) The effectiveness of the fire retardant is directly related to the moisture content of the dried product, i.e., to the severity of the drying operation.

Monoammonium phosphate is about twice as effective when the finished product is dried to a 7 to 8 percent moisture content compared with a product dried to 0.5 percent moisture. In both cases the samples were allowed to equilibrate to a moisture level of approximately 10 percent before flame testing. The relationship between flame spread and dried moisture content of the finished product is shown in figure 1. Monoammonium phosphate at the 15-percent level is used as an example. The flame spread rating shown on the vertical axis is an arbitrary scale depicting the advance of flame across the surface of the product.

These data clearly show a factor of 2 in flame rating between the nearly bone dry and 7- to 8-percent finished product moisture levels. The reason for this marked difference is not readily apparent. One can speculate that the cellulose begins to dehydrate when the moisture content of the product is low. This affects the hydroxyle groups on the cellulose in some irreversible way, thus decreasing the fire retardant efficiency.

Figure 2 shows the amount of fire retardant required to obtain various degrees of protection when the final product is dried to the 2-percent moisture level.

It can be seen that a flame rating of 40 is obtained when the product is treated with 10-percent MAP or DAP and dried to a 2-percent moisture level. If the retardant concentration were increased to about 22-

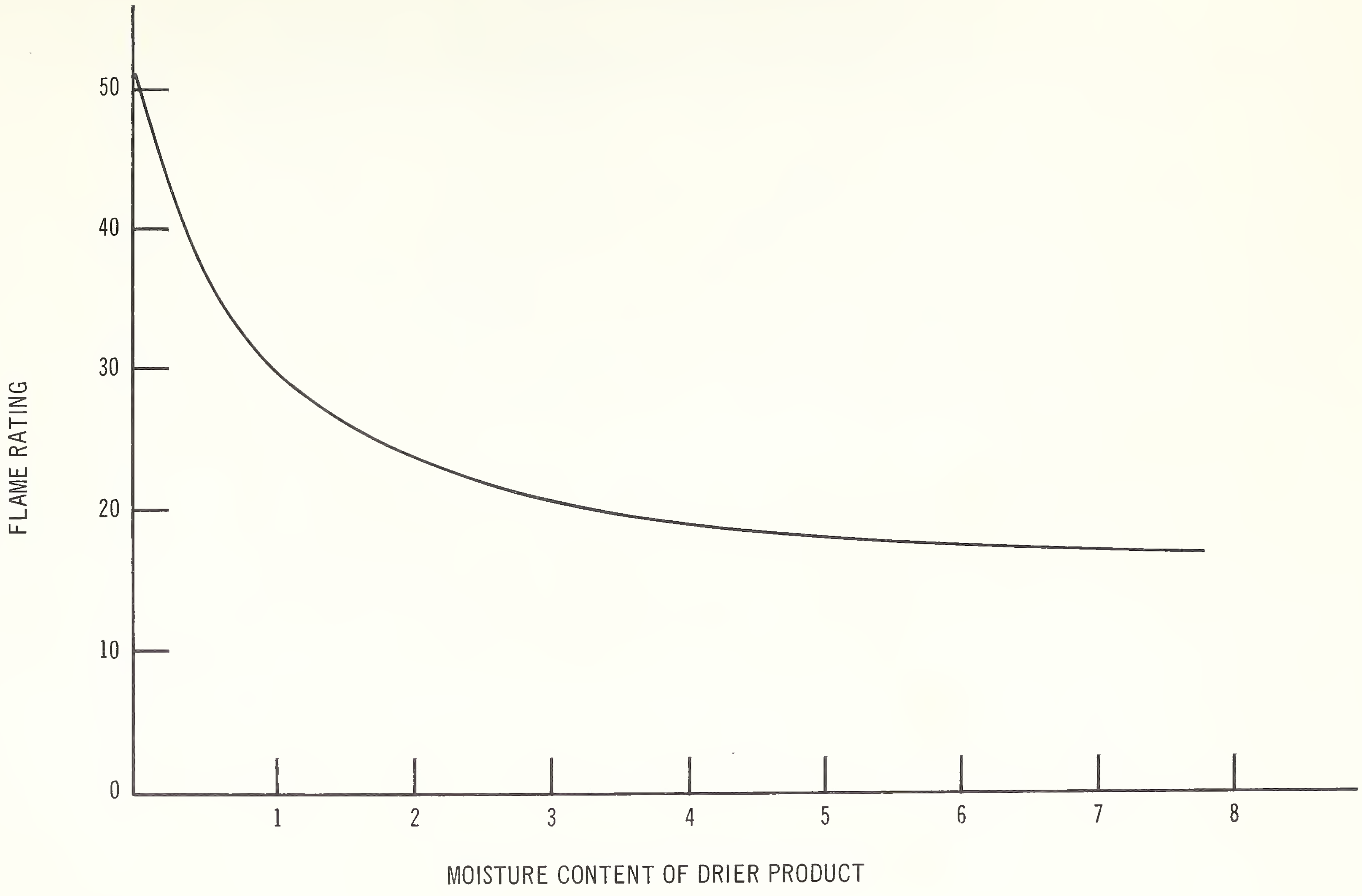

Figure 1.—Flame rating of monoammonium phosphate treated product after drying to various moisture levels.

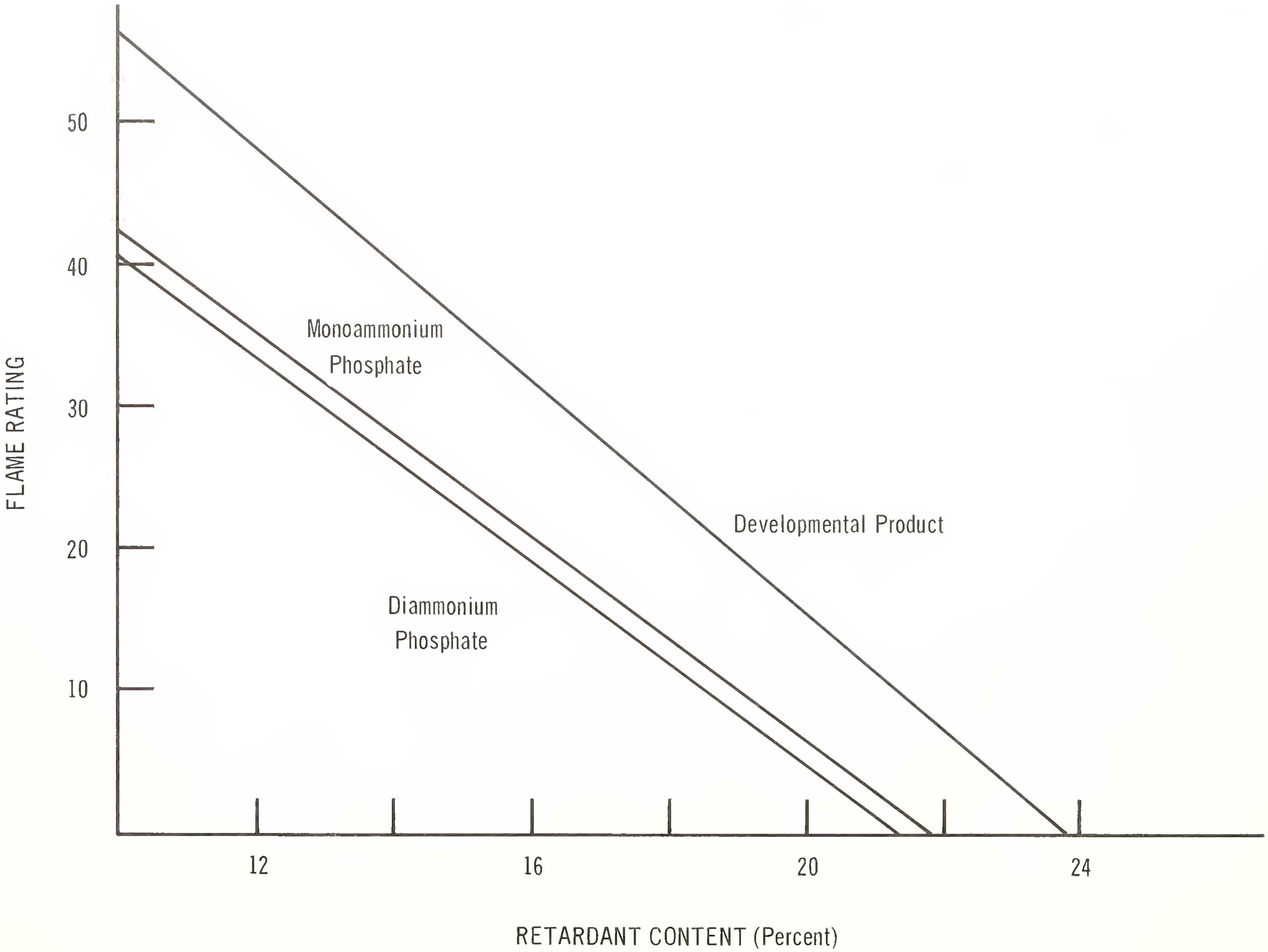

Figure 2.—Flame rating depression of retardant systems at 2 percent moisture level.

percent, the flame spread would be nearly zero. The developmental product, which is a modified ammonium phosphate, requires somewhat more fire retardant to obtain a degree of retardancy because it contains less phosphorus. The benefits of this product will be described later.

2) The second drying consideration arises from the fact that the physical properties of the retarded product degrade as the product is more thoroughly dried. This is illustrated in figure 3.

Higher surface temperatures can therefore be tolerated if the product is dried to a higher moisture level. The color rating is a measure of the degradation of the cellulosic fibers; a rating of 1 means essentially no color change and hence no damage. A color rating of 5 means severe damage such as fiber embrittlement and is evidenced by a dark brown-black color. The intermediate numbers represent increasing shades of tan to brown.

At a surface temperature of 320 ° F., it can be seen that a product dried to 8 percent moisture obtains an acceptable rating of 2, whereas at a 2 percent dried moisture level the rating is nearly 4. This would not be acceptable due to excessive degradation and subsequent loss of product integrity. In some cases the moisture content of the finished product is critical and a low value is required.

Figure 4 shows that a judicious choice of fire retardant can provide good flame ratings with little or no product degradation. In this work the product was again dried to a 2 percent moisture level. The developmental product causes only slightly more degradation than the untreated control over a fairly wide range of surface temperatures. Higher surface temperatures, of course, mean faster drying and more rapid throughput which would be desirable in most cases.

It is also interesting to note lowering of the characteristic decomposition point of cellulosic materials that occurs with fire retardant-treated materials. This is indicative of the beginning of the fire retarding reaction. Color rating is a measure of the progress of the fire-retardant reaction. The reaction nears completion when all black carbon is obtained at a color rating of 6.

The next application technique, spray treatment, may be of more interest since less additional equipment may be required. The following is a suggested spray procedure.

Spray treatment:

1. Prepare 30 percent solution of monoammonium phosphate
2. Spray web off garnett to obtain 50 percent add-on
3. Pass the lapped batting through squeeze rolls
4. Dry at a temperature not to exceed 270° F.
5. Dry to the maximum allowable moisture content

In this case, spraying the web as it comes off the garnett would be suggested. The MAP concentration is increased to 30 percent and thus only 50 percent wet weight add-on is required. The product would then be passed through squeeze rolls and dried as previously discussed. Alternate spray locations may be at the lapper or on the batt conveyor. It is conceivable that in some cases drying the finished batt may not be necessary.

A concentrated solution, used to add on 3 percent phosphorus, only increases the moisture content of the product by 6.4 percent, as shown below.

Spray treatment with liquid product:

P content	Wet add-on	Moisture added
Pct.	Pct.	Pct.
2	13	4.6
3	19	6.4
4	25	8.0

In general, we have found that 2 to 4 percent phosphorus is sufficient to impart fire-retardant properties to cellulosic materials and I arbitrarily chose 3 percent P as an example. Even less moisture is added, of course, if only 2 percent P is needed. (Previous workers have shown this to be a suitable level for fire retarding cotton batting.) The potential problem with spray application of a concentrated solution is the uniformity of treatment. The more uniform the treatment the better the performance.

The final application technique I would like to mention is roller coating. A roller coating device could be placed on the batting conveyor and used both to apply and squeeze the retardant into the fibers. Not much work has been done with this approach but it should have some merit.

Some work has been done with the dry application of powdered fire retardants. However, the losses of fire-retardant chemical during subsequent treatment of the fiber are high and the retention in the finished product is poor. We have concluded that some binder, such as the phenolic resins used to produce resinated cotton, will be necessary if water or some alternative carrying agent is excluded.

In conclusion, one must carefully consider the application technique and drying operation necessary to fire-retard cotton batting. It is hoped that the information presented in this paper will help in this regard.

A special report covering this topic is available. If a copy is desired please request Special Report No. 7225 from the author.

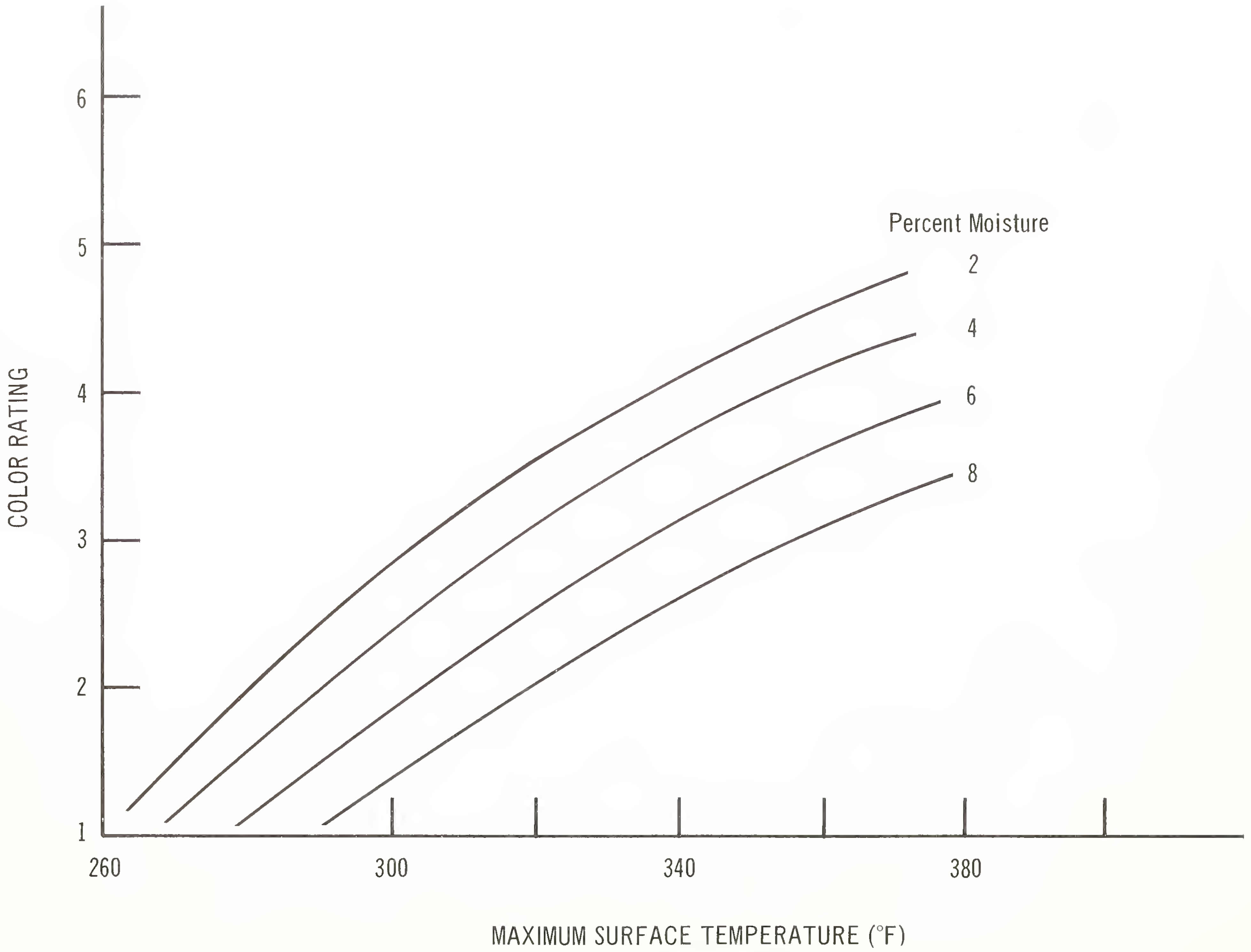

Figure 3.—The effect of moisture level and surface temperature on discoloration of MAP treated product.

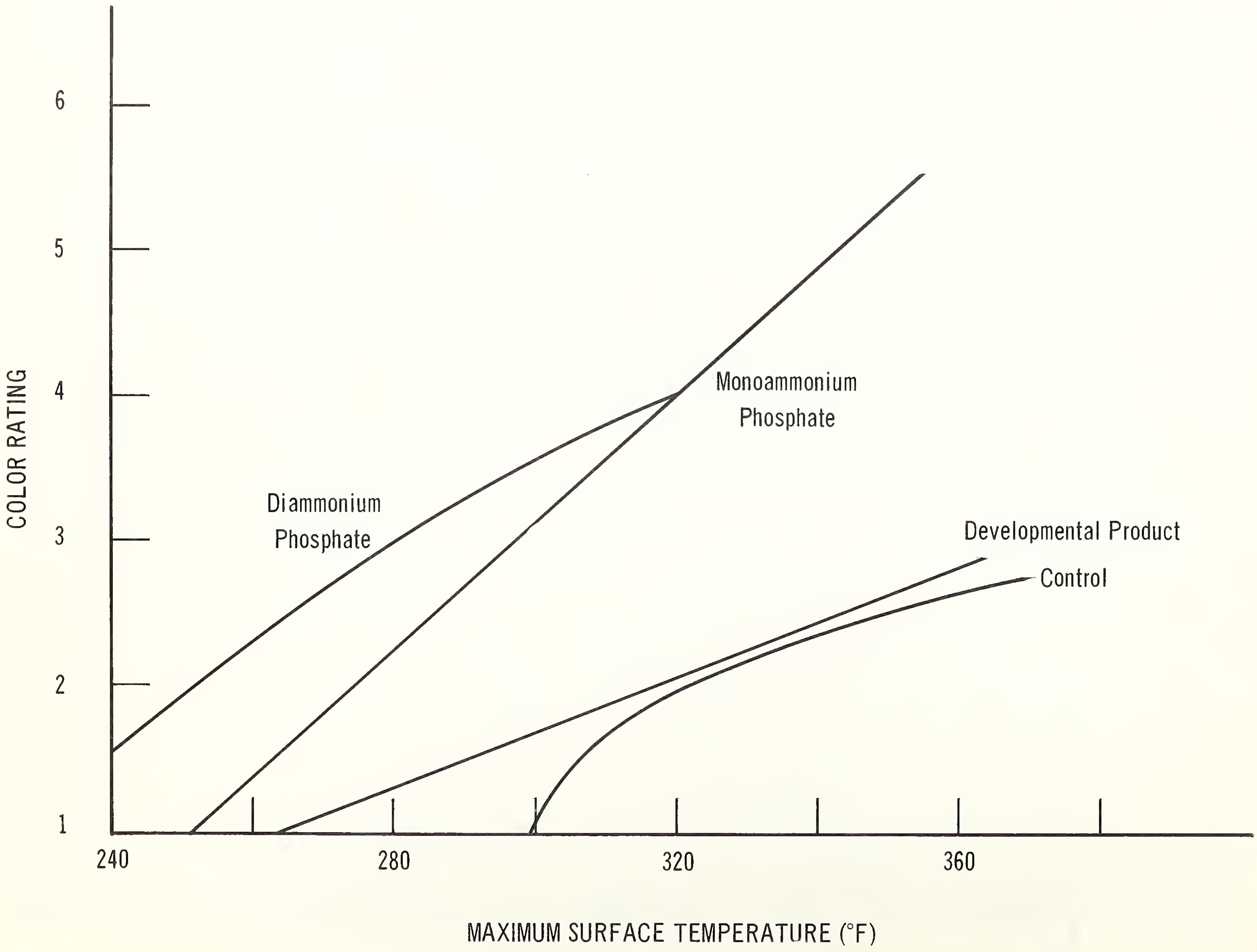

Figure 4.—The effect of maximum surface temperature on color or fire-retardant product.

CHLORINATED SOLVENT SYSTEMS FOR TEXTILE APPLICATIONS

by

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The Dow Chemical Company has developed a system called the *Dow Textile Solvent Process System* for use in textile finishing. This system employs chlorinated hydrocarbon solvents which have certain advantages over other organic solvents and water, such as:

1. Have low surface tension
2. Do not swell cellulose, but are excellent for wetting out cotton
3. Have no fire or flash point
4. Have low toxicity
5. Have low latent heat of vaporization (one tenth that of water)

The Dow Solvent System, which has been designed for continuous processing of woven textiles, has the following advantages over aqueous processing equipment:

1. The drying cost is lower because of the lower heat of vaporization of chlorinated solvents.
2. It requires less space. (The entire processing unit minus materials handling equipment is 7 feet by 7 feet.)
3. It reduces utilities costs.
4. There is no pollution problem because by a properly operated unit no chemicals are discharged into rivers or lakes and no vapors are vented to the atmosphere.
5. It is usable for both natural and synthetic fibers.

An open loop system (in which water is brought into the plant and then discarded after it is used) is typical of most aqueous textile processes. Early nonaqueous solvent processes were also open loop with the solvent vapors being vented to the atmosphere. The Dow Textile Solvent Process System employs a highly efficient closed loop system. Fabric is treated with the chlorinated solvent solution; the solvent is then removed in a recovery system; the water in the recovered solvent is removed in a separator, and the reclaimed solvent is placed into a storage tank for later use in preparing more treating solution—thus, almost no solvent is lost, making the use of chlorinated solvents economical and safe.

The Dow Textile Solvent Process unit consists of two parts, an application module and a solvent recovery module. The two modules are connected in operation, but are easily separated so that more than one application method can be used with the same solvent recovery module. The units now in operation all use a spraying system, since this method has been entirely adequate. However, immersion, pad, and knife coating systems are feasible. It would require 1 to 12 months to develop these methods, depending on whether available commercial units can be adapted or if wholly new systems must be designed and built.

After resin application, the wet fabric passes into the solvent recovery module, which is filled with heavier-than-air chlorinated solvent vapor. This vapor permeates the fabric and wets it even further, thus aiding the distribution of chemicals from selvedge to selvedge. The fabric passes over steam heated drying cans which are held above the boiling temperature of the solvent being used. The solvent is vaporized, and the fabric emerges dry and solvent free from the recovery unit. The additional solvent vapor produced during evaporation from the wet fabric is condensed on cooling coils that encircle the upper part of the recovery unit, and this condensed solvent is passed on the water separator and storage tanks.

The use of heated cans has been adequate in our present commercial applications, but we realize that contact with hot metal surfaces during solvent removal is unacceptable for some applications, such as dyeing. Therefore, we have studied other drying methods, and are incorporating them in some of our newer equipment.

The following running parameters may indicate equipment:

20 yards per minute
7 hour day
5 day week
20 day month
12 ounce fabric

These parameters give a capacity of 1,200 yards per hour, or 8,400 yards per day, or 168,000 yards per month (or 126,000 pounds per month), or 2 million yards per year (or 1.5 million pounds per year).

The present commercial applications for the Dow units include wood stabilization, fluorochemical stain repellence, waterproofing, and fabric modification through softeners, etc. The applications now in the development stage include flame retardancy, high-speed dyeing, fabric backings, and durable press (including soil release). The applications now in the research stage include sizing, desizing, scouring, bleaching, and other areas of finishing not already in the commercial or developmental stages. By adding up the applications in the commercial, developmental and research stages, one can readily see that the Dow effort is directed toward the complete replacement of water by chlorinated solvents in textile processing.

These, then, are the technical features of the Dow Textile Solvent Process system. For your further information about the system, I will describe briefly its financial aspects. Dow uses a 3-year service contract arrangement with the customer in which the customer pays a monthly service charge based on pounds of fabric processed; the customer is thus not required to make a large capital investment. Under this service contract, Dow provides:

1. The application and solvent recovery modules
2. All instrumentation, pumps, and mixing and storage tanks
3. All major maintenance parts
4. All solvents for mixes and applications
5. Technical service to insure performance and safety
6. An operator training program

For his part, the customer provides the materials handling equipment, the utilities, the finishes, an operator, and the installation labor. At present the lowest monthly minimum charge is $3,000, for a guaranteed 75,000 pounds of fabric at 4¢ per pound; a charge of 37½¢ per pound is then charged for each pound above the minimum quantity. A sliding scale is used so that if the customer can guarantee 300,000 pounds of fabric per month, $6,000 per month minimum is charged, or 2¢ per pound, and 1¢ per pound for each pound above the minimum.

A smaller pilot plant unit is available for sale to the customer who wishes to do solvent research and development with no strings attached. This unit is about 4½ by 4½ feet in size, and can handle fabric widths up to 18 inches. A full sized commercial unit is available in Midland, Mich., for use by any textile processor or chemicals supplier who wishes to evaluate this textile process under commercial conditions; these trails involve no commitment to Dow.

We have had some experience in the treating of raw cotton (before garnetting) with chlorinated solvent solutions of resins. However, the equipment used differed from that presently being used for fabrics. We would expect that a modified unit that does not use heated cans for drying would be quite suitable for the treatment of raw cotton or batting.

Dow has had a full-time research program on textile flame retardancy for over 7 years. Most of our previous effort was concentrated on tris (1-aziridinyl) -phosphine oxide (APO). Now that we are selling our APO technology to a smaller chemical firm, we are investigating a variety of other flame retardant chemicals for use in both aqueous and nonaqueous media, but with somewhat greater emphasis on chlorinated solvent soluble materials. We are looking at both nonreactive and reactive monomers and polymers that contain phosphorus.

A byproduct of our research effort has been the development of a new flame retardancy screening method and device. We do have permission to describe the equipment or the principle on which it works, but we can describe some of its features. This method involves the application of chemicals to small samples which are then burned and evaluated on the basis of how the burning compares with that of non-treated samples. It is especially useful when evaluating a new compound that is available in a very small quantity, for only a few grams are necessary rather than the larger quantities usually needed. By this method one can determine ignition time, the temperature of the combustion gases, total burning time, and the presence and duration of afterglow. We have used it primarily as a screening method for comparing the flame-retardant efficiencies of different chemical compounds on varying substrates, but some of our people are interested in trying to get it adopted as a government approved standard test method. Dow has filed a patent case covering the method and equipment, but is planning to sell the manufacturing rights. One manufacturer is now preparing to produce these devices, and further information can be obtained from W. C. Weaver of Dow's Technical Service and Development Department in Midland, Mich.

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